

AMERICAN CHEMICAL SOCIETY DIVISION OF PESTICIDE CHEMISTRY

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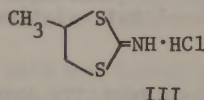
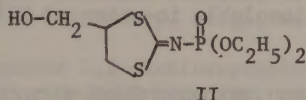
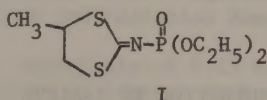
MONDAY MORNING - GENERAL - C. H. Van Middeltem, Presiding

1. DEGRADATION OF ETHYLENETHIOUREA IN SOIL. D. D. Kaufman and C. L. Fletcher, Pesticide Degradation Laboratory, Agricultural Environmental Quality Institute, ARS, U.S. Department of Agriculture, Beltsville, Md. 20705.

^{14}C -Ethylene-labeled ethylenethiourea (ETU) was used to examine the persistence and degradation of ETU in autoclaved and nonsterile Lakeland sandy loam and Hagerstown silty clay loam. Chemical analysis of ^{14}C -ETU treated soils revealed that all of the ETU was converted to 2'-imidazolidone within 2 days in soils treated with 2 and 20 ppm ETU, and within 8 days in soil receiving 200 ppm. A much slower conversion of ETU to 2'-imidazolidone also occurred in autoclaved soil. Further degradation occurred rapidly in non-sterile Hagerstown soil: 43.4, 8.9, and 0.9% of the ^{14}C was evolved as $^{14}\text{CO}_2$ within 4 days after treatment with 2, 20, and 200 ppm ^{14}C -ETU, respectively. A similar, but somewhat slower degradation pattern was observed in the Lakeland soil. Essentially no $^{14}\text{CO}_2$ was evolved from autoclaved soils. Very little degradation of ETU was observed in soil enrichment cultures containing 50 ppm ETU. Rapid degradation of ^{14}C -ETU to $^{14}\text{CO}_2$ was observed in Fenton's reagent, a free radical generating system. The role of chemical and biological factors in the degradation of ETU in soil will be discussed.

2. CYTROLANE[®] SYSTEMIC INSECTICIDE: METABOLISM IN COTTON PLANTS. J. Zulalian American Cyanamid Company, P. O. Box 400, Princeton, N.J. 08540.

The metabolism of CYTROLANE Systemic Insecticide, cyclic propylene (diethoxyphosphinyl)dithioimidocarbonate (I), labeled with carbon-14 in the imino carbon atom, was investigated in cotton plants. CYTROLANE was identified as the predominant residue sixty days after foliar application of the radiotracer. A number of water-soluble metabolites were found of which four were considered major metabolites and three were minor. One major metabolite was identified as radiothiocyanate ion. Hydrolysis of two major metabolites by β -glucosidase liberated two closely related compounds which were isolated by thin-layer chromatography. One of these was identified as cyclic (hydroxymethyl)ethylene (diethoxyphosphinyl)dithioimidocarbonate (II) and analysis of the other aglycone by chemical ionization-mass spectrometry gave a peak at $M=283$ for the aglycone. Two of the minor metabolites of CYTROLANE were also derived from the metabolism of carbon-14-labeled cyclic propylene dithioimidocarbonate hydrochloride (III), the synthetic intermediate to CYTROLANE. Therefore, these metabolites do not retain the diethylphosphinyl moiety of CYTROLANE and may not be of toxicological significance.



3. THE EFFECT OF ISOPROPYL-3-CHLOROCARBANILATE AND ITS PLANT METABOLITES UPON MITOCHONDRIAL OXIDATIVE PHOSPHORYLATION. Gerald G. Still and Donald G. Rusness, Agricultural Research Service, U. S. Department of Agriculture, Metabolism and Radiation Research Laboratory, Fargo, North Dakota 58102.

The effects of the herbicide, isopropyl-3-chlorocarbanilate, and its hydroxylated metabolites, isopropyl-5-chloro-2-hydroxycarbanilate and isopropyl-3-chloro-4-hydroxycarbanilate, upon NADH oxidation, P_i uptake or release, and ATP formation were studied in corn mitochondria. The results indicated that 0.1 mM isopropyl-3-chlorocarbanilate

and the 2-hydroxy-metabolite inhibited NADH oxidation by 30% whereas only the 2-hydroxy-metabolite inhibited NADH-linked ATP formation (85-100%). Dinitrophenol and the 2-hydroxy-metabolite exerted similar effects upon respiration, phosphorylation, and ATPase activity. The 4-hydroxy-metabolite (0.1 mM) exerted no effect upon respiration, phosphorylation, or ATPase activity. The β -O-glucoside conjugates of the hydroxy-metabolites of isopropyl-3-chlorocarbanilate did not inhibit NADH-linked respiration or phosphorylation at 0.1 mM concentrations. Comparative studies with corn, cucumber, and soybean mitochondria indicated that the parent herbicide and its metabolites affected respiration and phosphorylation activities in a similar manner. These data will be discussed in terms of plant resistance and susceptibility to isopropyl-3-chlorocarbanilate.

4. MASS SPECTRAL CHARACTERIZATION OF THE SHEEP URINARY METABOLITES FROM RUELENE. ^(A)

Jerome E. Bakke and Connie E. Price, U. S. Department of Agriculture, Agricultural Research Service, Metabolism and Radiation Research Laboratory, Fargo, N. Dak. 58102.

Eleven sheep urinary metabolites from ^{14}C -Rulene (4-*tert*-butyl-2-chlorophenyl methyl methylphosphoramidate) were either identified or characterized by mass spectrometry of their trimethylsilyl and/or the trimethylsilyl derivatives of their methyl esters. The identified metabolites and approximate % of the urinary ^{14}C were as follows: ($\emptyset$ = 2-chloro-*p*-phenylene)

I. $(\text{CH}_3)_3\text{C}\emptyset\text{OH}$	0.4%
II. $(\text{CH}_3)_3\text{C}\emptyset\text{OP}(\text{O})\text{OH}(\text{OH})$	1.1

Those characterized by mass spectrometry were:

III. $\text{HOCH}_2(\text{CH}_3)_2\text{C}\emptyset\text{OH}$	6.6
IV. $\text{HOOC}(\text{CH}_3)_2\text{C}\emptyset\text{OH}$	5.0
V. $\text{HOCH}_2(\text{CH}_3)_2\text{C}\emptyset\text{OP}(\text{O})\text{OH}(\text{OH})$	10.0
VI. $\text{HOCH}_2(\text{CH}_3)_2\text{C}\emptyset\text{OP}(\text{O})\text{OCH}_3(\text{NH}_2)$	3.0
VII. $\text{HOCH}_2(\text{CH}_3)_2\text{C}\emptyset\text{OP}(\text{O})\text{OH}(\text{OCH}_3)$	15.0
VIII. I glucuronide	40.0
IX. III glucuronide (aromatic) major	
X. III glucuronide (aliphatic)	
XI. VII glucuronide (position unknown)	

In addition, metabolites representing 8% of the urinary activity appeared to be Rulene that had been oxidized to two different aldehydes.

5. SYNTHESIS OF GLUCOSIDES OF ISOPROPYL 3-CHLOROCARBANILATE METABOLITES. George G. Ecke, Chemical Division, PPG Industries, P.O. Box 31, Barberton, Ohio 44203.

Soybean plants growing in a nutrient solution containing isopropyl 3-chlorocarbanilate have been shown to contain two conjugates, which upon hydrolysis yielded the aglucones, isopropyl 5-chloro-2-hydroxycarbanilate (I) and isopropyl 3-chloro-4-hydroxycarbanilate (II) (G. G. Still and E. R. Mansager, Div. of Pesticide Chemistry, 164th National Meeting ACS, August 1972). The O- β -D-glucosides of I and II have been synthesized for use in analytical studies. Reaction of I with tetra-O-acetyl- α -D-glucosyl bromide and sodium hydroxide in aqueous acetone gave 29% yield of the tetraacetate of the glucoside. Deacylation with methanol and a catalytic amount of sodium methoxide gave 86% of the glucoside of I. Similarly treating II yielded 32% of the tetraacetate, which was deacylated to yield 86% of the glucoside of II. The Koenig-Knorr and the Helferich reaction both gave results inferior to the above procedure. Both glucosides were very soluble in methanol but relatively insoluble in water at 25°.

6. CORRELATIONS OF TOXICITY AND ACETYLCHOLINESTERASE (AChE) INHIBITION IN 2-ALKYL SUBSTITUTED 1,3-BENZODIOXOLYL-4 N-METHYLCARBAMATES AND RELATED COMPOUNDS. Chieh-Yu, Illinois Natural History Survey, Urbana, Illinois, 61801.

2-Alkyl substituted-1,3-benzodioxolyl-4 and 1,3-benzodioxolyl-5 N-methylcarbamates, 2-methyl substituted indanyl-4 and indanyl-5 N-methylcarbamates, and 2-methyl substituted quinolinyl-8 N-methylcarbamates were examined in this study. Oral toxicity (LD₅₀) to female mice and topical LD₅₀ values to the house fly were determined. Main's kinetic constant's (affinity, carbamylation and over-all inhibition rate) on AChE were also determined. There were good correlations between the *in vivo* toxicity to mice (without synergist) or house fly (with synergist) and the *in vitro* over-all enzyme inhibition rate. Increasing the number or the size of the alkyl substitution (i.e., unsubstituted

vs monomethyl, monomethyl vs dimethyl or dimethyl vs diethyl) increased the toxicity and the over-all enzyme inhibition. The increase in the enzyme inhibition by substitution was mainly due to the increase in the affinity of the enzyme-inhibitor complex. It is interpreted that the substituted alkyl groups interact with the anionic or hydrophobic site of the AChE through van der Waal's forces or hydrophobic bonding. The enzyme inhibition and toxicity were maximal when the second ring of these bicyclic compounds was attached in the 2,3-position in relation to the hydroxyl group of the phenyl ring.

7. ISOLATION OF ACYLANILIDE-HYDROLYZING MICROBIAL ENZYMES. Joan Blake and Donald D. Kaufman, Pesticide Degradation Laboratory, Agricultural Environmental Quality Institute, ARS, U.S. Department of Agriculture, Beltsville, Md. 20705.

Two acylanilide-hydrolyzing enzymes were isolated from the soil fungus Fusarium oxysporum Schlecht. Although one enzyme was induced by propanil and the other by p-chlorophenyl N-methylcarbamate (PCMC), both enzymes actively hydrolyzed 3',4'-dichloropropionanilide (propanil) to 3,4-dichloroaniline and propionic acid. Neither enzyme showed any activity on PCMC. Certain methylcarbamates are known to inhibit acylanilide- and phenylcarbamate-hydrolyzing enzymes of soil microorganisms. Propanil hydrolysis by both enzymes was inhibited by PCMC. Although similar in function, the enzymes differed in specific activity, substrate specificity, relative stability, and pH optima. The role of PCMC as an inducer and inhibitor of acylanilide-hydrolyzing enzymes will be discussed.

8. THE PROPERTIES OF AN ARYL ACYLAMIDASE FROM DANDELION WHICH HYDROLYZES 3',4'-DICHLOROPROPIONANILIDE. Robert E. Hoagland, E. D. Handel and G. Graf, Department of Biochemistry, North Dakota State University, Fargo, North Dakota 58102

Several plant enzymes which are able to hydrolyze the herbicide, propanil (3',4'-dichloropropionanilide), have been reported [R.E. Hoagland and G. Graf, Phytochem. **11**, 521 (1972); R.E. Hoagland and G. Graf, Weed Sci. **20**, 303 (1972); D.S. Frear and G. G. Still, Phytochem. **7**, 913 (1968)]. Another plant aryl acylamidase which hydrolyzes propanil has been isolated from dandelion roots (Taraxacum officinale Weber). Substrate specificity tests on the partially purified enzyme revealed a broad substrate preference. Several chlorinated ring-substituted propionanilide analogs such as 4'-chloropropionanilide, 3'-chloropropionanilide, 2'-chloropropionanilide, 2',4'-dichloropropionanilide and 2',3'-dichloropropionanilide were hydrolyzed. Hydrolysis was also found on other analogs of propanil such as 3,4-dichloroacetanilide, 3',4'-dichlorobutyranilide and 3',4'-dichlorovaleranilide. Amide bonds in the urea herbicides, monuron and fenuron, and in the carbamates, propham and chlorpropham, were not hydrolyzed. The enzyme was inhibited by sulfhydryl reagents and several metal ions. Temperature studies indicated that the enzyme was relatively stable up to 43°C but lost activity rapidly when exposed to higher temperatures. The pH optimum was between 7.4 - 7.8. A Lineweaver-Burk plot yielded an apparent K_m of $1.7 \times 10^{-4}M$ with 3',4'-dichloropropionanilide. The properties of this enzyme will be compared to those of the other plant aryl acylamidases which hydrolyze propanil.

9. PHOTOLYSIS OF 2,2-DICHLOROPROPIONIC ACID IN AQUEOUS SOLUTION. Fred S. Tanaka and R. G. Wien. Agricultural Research Service, U. S. Department of Agriculture, Metabolism and Radiation Research Laboratory, Fargo, North Dakota 58102.

The photolysis of 0.25 M aqueous 2,2-dichloropropionic acid was investigated using 253.7 nm light. Carbon dioxide, chloride ion, pyruvic acid, acetaldehyde, and 1,1-dichloroethane were identified as degradation products. A water insoluble polymeric product was also observed and partially characterized. Quantum yields of the photolysis products were determined for agitated samples flushed with either air or nitrogen and nonagitated samples sealed in the presence of air. The per cent yield for 2,2-dichloropropionic acid (unreacted) and most of the photolytic products was estimated as a function of irradiation time. A comparison of the 2,2-dichloropropionic acid quantum yields under nonoxygenated conditions was made with data previously reported for dichloroacetic acid, and the results indicate that dichloropropionic acid is more easily photodegraded. Therefore, the additional methyl group in the next higher homolog does appear to affect the photochemistry of the 2,2-dichloroinated acids.

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10. METABOLISM, EXCRETION AND STORAGE OF HEXACHLOROBENZENE IN THE MALE RAT. H. M. Mehendale, H. B. Matthews, National Institute of Environmental Health Sciences, P. O. Box 12233, Research Triangle Park, N. C. 27709.

In addition to its use as a fungicide for seed-coating, hexachlorobenzene (HCB) is also used industrially in organic synthesis. It causes cutaneous porphyria characterized by skin rashes and extreme sensitivity to light. Very little is known about its metabolism and existing reports indicate that it is not metabolized. Uniformly labeled HCB-¹⁴C was administered to male rats as a single oral dose in Arachis oil. Less than 20% of the administered dose was excreted after 7 days. Urinary excretion was less than 1% of the total dose. Examination of the excretory pattern indicates that excretion will cease by 3 to 4 weeks and over 90% of what is stored at the end of 7 days would be retained in the animal body. Fat, muscle, liver, and small intestines contained most of the HCB-¹⁴C stored in the body. Every tissue examined contained HCB and at least one dechlorinated metabolite. Urine contained at least 7 metabolites including pentachlorophenol, pentachlorobenzene, and tetrachlorohydroquinone. Fecal extractions gave only one radioactive spot which corresponded to HCB. Liver, lung, kidney and intestinal enzyme preparations metabolize HCB predominantly to dechlorinated products. These metabolites are produced by microsomal preparations and NADPH is not required for their formation. In the presence of NADPH, microsomal preparations produce pentachlorophenol. In the liver addition of UDPGA to microsomal incubations along with NADPH results in the disappearance of pentachlorophenol from the organic extracts and the corresponding radioactivity appears in the aqueous phase. In contrast, kidney preparations fail to respond to UDPGA.

11. THE PERSISTENCE OF DIELDRIN AND HEPTACHLOR IN A FIELD SOIL.

H. P. Freeman, A. W. Taylor, Agricultural Research Center, ARS, USDA, Beltsville, Maryland 20705, and W. M. Edwards, ARS, USDA, Coshocton, Ohio 43812.

In 1966, Dieldrin was incorporated at 5.6 kg/hectare to the 7.5 cm depth on an 1.09 hectare watershed with a silt loam soil immediately before maize was planted. Samples were taken from subsections of the watershed at regular intervals until October 1972 and analyzed for dieldrin residues. The rate of loss of dieldrin was described by the linear regression $D = 2.76 - 0.224T$, where D is in ppm and T in years. The best estimate for total disappearance of the dieldrin is 12 years, with a 90% probability that the true value is between 6.5 years and about a century. Although the results are confounded by sample variability there is evidence that the most probable disappearance time varies between these limits at different places on the watershed, depending on the soil moisture regime. In a similar experiment begun in 1969 on a similar soil, the rate of loss of heptachlor was found to follow the equation $H = 242 - 95.4 T$ where H is in mg/m² of field area, corresponding to a most probable disappearance time of 2.5 years. No degradation products of dieldrin were found, but γ chlordane, hydroxychlordane and heptachlor epoxide were found in the heptachlor samples.

MONDAY AFTERNOON - PANEL DISCUSSION: PR70-15 ENVIRONMENTAL IMPACT REQUIREMENTS - R. A. Conkin, Presiding

12. ENVIRONMENTAL ASPECTS OF PESTICIDE REGISTRATION. Carroll W. Collier, U. S. Environmental Protection Agency, Pesticides Regulation Division, Washington, D. C. 20250.

Environmental concern over pesticides is reflected in PR Notice 70-15, dated June 23, 1970. The intent of this notice is still considered applicable to the registration of pesticide products. PR Notice 70-15 deals with the following areas of environmental concern:

1. Rate of dissipation of the pesticide and its degradation products in soil.
2. Mechanism of degradation of pesticide residues in soil, sediment and water.
3. Leaching of pesticide residues.
4. Runoff studies.
5. "Bound" residues.
6. Bioaccumulation and the dosage effect on aquatic and terrestrial organisms.

Information generated in these 6 areas will normally allow for consideration of the fate, movement and effect of a pesticide in the environment. The amount of

information required for a pesticide is partially dependent on the proposed pattern and extent of use. Additional interpretation of data requirements considered responsive to PR Notice 70-15 will be available in a forthcoming guideline entitled "Guidelines for the Registration of Pesticides," Section IV.

13. A PROPOSED APPROACH TO PR 70-15 GUIDELINES FOR STUDIES TO DETERMINE THE IMPACT OF PESTICIDES ON THE ENVIRONMENT. C. A. I. Goring, J. W. Hamaker, D. A. Laskowski, Dow Chemical U.S.A., 2800 Mitchell Drive, Walnut Creek, California - 94598.

The critical objectives of environmental research on new pesticides are defined and discussed. The problems involved in accomplishing these objectives are examined and analyzed, and a specific plan of action is proposed. The program suggested involves the determination of key behavioral characteristics of new pesticides under controlled, standardized conditions and comparison of these characteristics with those of established "benchmark" pesticides whose behavior and impact in the environment are known. The expected behavior and potential impact of the new pesticide in the environment may then be predicted. The need to develop perspective on the comparative behavior and impact of pesticides and naturally occurring chemicals in the environment is briefly discussed.

14. ONE INDUSTRIAL VIEW OF NOTICE PR 70-15. John E. Boyd, Agricultural Division American Cyanamid Company, Princeton, New Jersey, 08540.

Some of the most difficult problems involved in laying the foundation for protection of the environment from deleterious contamination with pest-control agents are not technical in nature, but lie rather in the design of the meaningful experiments which are technically and economically feasible and which are reasonably interpretable in terms of commercial-scale operation. Particularly subject to misinterpretation and/or over interpretation are the results of radiotracer experiments which, by their nature, must be conducted on a small scale and under quite artificial conditions. The data which can be most validly extrapolated to commercial-scale operations are those obtained by field residue studies, conducted under actual use conditions, in which environmental samples, such as soil, water, etc., are collected and analyzed by conventional residue methods developed especially for the sample types at hand. These methods must, of course, be supported by "metabolism" data adequate to show that the analytical results obtained are truly representative of the total potentially deleterious residues which may be present in the samples. While most of the requirements listed in Notice PR 70-15 are considered to be valid concerns of EPA, several are primarily of academic interest and, in our opinion, have little place in this list. A compilation of detailed acceptable protocols is needed from EPA to avoid the unnecessary costs of the confusion and wasted motion which characterize the present situation.

15. AN ACADEMIC LOOK AT ENVIRONMENTAL REGULATORY REQUIREMENTS. Robert L. Metcalf, University of Illinois, Urbana-Champaign, Illinois 61801.

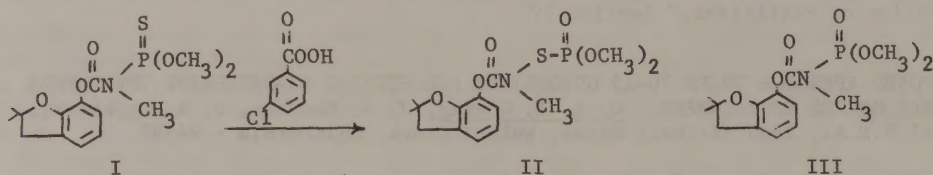
New environmental regulatory requirements for pesticides represent excursions into areas of biology, biochemistry, and analytical chemistry well beyond the conventional. Nevertheless this approach is essential if we are to preserve a reasonable sort of environmental quality. Biological short-cuts to the determination of the environmental impact of candidate pesticides are under development and have already provided information useful to state, national, and international agencies. Sample studies of this sort will be reviewed in detail.

TUESDAY MORNING - SECTION A - SYMPOSIUM ON NEW ASPECTS OF ORGANOPHOSPHORUS PESTICIDES - T. R. Fukuto, Presiding

16. OXIDATIVE REARRANGEMENT OF ORGANOPHOSPHORUS THIONATE ESTERS. David A. Wustner, T. R. Fukuto, and M. A. H. Fahmy, Department of Entomology, Division of Toxicology and Physiology, University of California, Riverside, Calif. 92502.

2,2-Dimethyl-2,3-dihydrobenzofuran-7 N-methyl-N-(dimethoxyphosphinothioyl) carbamate (I) is a highly effective selective insecticide, i.e., it is toxic to insects but is substantially less toxic to mammals. The basis for its selective toxicity appears to reside in the ability of insects to metabolize I to the highly toxic carbofuran more efficiently than mammals by means of oxidative and hydrolytic processes. In our study

of the oxidation of I by *m*-chloroperbenzoic acid, we have discovered that I oxidatively rearranges to the corresponding *N*-dimethoxyphosphinylthio derivative (II), in addition to direct desulfuration to the expected *N*-dimethoxyphosphinyl derivative (III).



A similar oxidative rearrangement was observed in the peracid oxidation of Dyfonate[®] (O-ethyl *S*-phenyl ethylphosphonodithioate) yielding phenyl ethoxy(ethyl)phosphinyl disulfide. A proposed mechanism for this rearrangement will be discussed.

17. NEWER ASPECTS OF METABOLISM OF PHOSPHONATE INSECTICIDES. Julius J. Menn, J. Bruce McBain, Stauffer Chemical Co., Agricultural Research Center, P. O. Box 760, Mountain View, California 94040.

Phosphonate insecticides are recognized as organophosphorus esters containing one P-C bond. In this category are usually included: phosphonate, phosphonothioate, and phosphonodithioate esters. Although some of the toxicological properties of phosphonates were first described by Schrader and his associates 35 years ago [Mon. 62, 3rd ed., Verlag Chemie GmbH, Weinheim/Bergstr. (1963)], their insecticidal potential has been reduced to practice on a broad scale only in recent years. Relatively little information is available on the metabolic fate of phosphonate insecticides in plants and animals [Pure and Appl. Chem. Supplmt. 57 (1971)]. This paper discusses metabolic data which indicate that the P-C bond in these molecules remains intact in the course of biotransformations in plants and animals. As a rule these terminal metabolites consist of alkyl or aryl phosphonothioic or phosphonic acids. These acids are relatively non-toxic, do not accumulate in tissues, and are readily excreted.

18. ORGANOPHOSPHORUS INSECT CHEMOSTERILANTS. Alexej B. Bořkovec, U.S. Department of Agriculture, ARC, Beltsville, Maryland 20705.

Alkylating derivatives of phosphoric and phosphorothioic acids and cyclic azaphosphorines are chemical cytostatic and mutagenic agents with selective activity on gonadal tissues of insects. Structure-activity relationship studies revealed a primary role of the alkylating moiety and secondary role of the phosphorus carrier moiety. Toxicological properties of this class of chemosterilants are basically different from those of phosphorus insecticides. Nonalkylating organophosphorus chemosterilants show similarities in mode of action that implicate possible activation step as a condition for biological activity. Structure-activity correlations indicate species specific differences in metabolic patterns and much narrower range of activity as compared with the alkylating analogs. Experiments with synergists and with resistant strains of insects revealed some basic differences between the biological activity of phosphorus chemosterilants and insecticides.

19. STRUCTURE AND BIOACTIVITY OF ORTHENE ANALOGS. Philip S. Magee, Ortho Division, Chevron Chemical Company, Richmond, California. 94804.

MONITOR (O,S-dimethyl phosphoramidothiolate) and its N-acetyl analog, ORTHENE, represent an important new class of insecticides, structurally different from older types of phosphates. Extensive studies in the synthesis of ORTHENE analogs by three different methods were successful in providing all of the desired structures. These include variations in chain length, branching, unsaturation and various hetero and halo substitutions in the acyl group. Some of the by-product chemistry is interesting, and the physical properties of the analogs were unique enough to prove important in their separation from other cholinergic materials.

As structure is changed, many unusual variations in bio-activity (mammalian toxicity and insecticidal activity) are displayed, especially when compared with related MONITOR analogs. Indeed, what appears to be simple acyl modifications of MONITOR-class insecticides show markedly different responses to identical changes in structure. Among the most interesting observations is the extreme sensitivity of bioactivity

to minor changes in O and S substitution, which do not greatly affect MONITOR activity. Adverse effects are also apparent in acyl chain branching or cyclization despite the remarkable retention of high activity out to 18 carbon atoms.

20. STRUCTURE AND FUNGITOXICITY OF ORGANOPHOSPHORUS COMPOUNDS.

Henry Tolkmith, Dorsey Mussell, Dow Chemical U.S.A., Midland, Michigan 48640.

A number of organic phosphorus compounds have been found to be active against fungi pathogenic for agriculturally important plants. Such compounds represent two separate classes of structure. One class comprises substances containing at least one sulfur atom attached directly at phosphorus. Organophosphorus fungicides of this kind belong to several types of compounds, viz., phosphonothioates, phosphorothiolates and phosphorothiolothionates. The other class comprises products that are phosphorus derivatives of N-heterocyclics, such as maleimide, imidazole, triazole and pyrazolopyrimidine. The antifungal activity and mammalian toxicity of the compounds indicated will be discussed.

TUESDAY MORNING - SECTION B - GENERAL - H. F. Enos, Presiding

21. VAPOR-PHASE PHOTODECOMPOSITION OF *p,p'*-DDT AND ITS RELATIVES. Kenneth W. Moilanen, and Donald G. Crosby, Department of Environmental Toxicology, University of California, Davis, Calif. 95616.

Investigations with the vapor-phase photoreactor described previously (Moilanen and Crosby, ACS Meeting, Boston, 1972) have been extended to the decomposition of DDT and its relatives. DDT was converted to DDE which underwent further decomposition to four electron-capturing, chlorinated photoproducts. In addition, 3,6-dichlorofluorenone was detected as a product of DDT photodecomposition taking place on the reactor walls. Several improvements in the photoreactor, including a new source of ultraviolet light, have been directed toward closer simulation of environmental conditions.

22. INVESTIGATION FOR BOUND RESIDUES IN TISSUES FROM CATTLE FED 2,4,5-T D. J. Jensen, P. W. Miller, L. F. Berhenke, Agricultural Department, Dow Chemical U.S.A., P. O. Box 1706, Midland, Michigan 48640.

Muscle, fat, liver, and kidney from animals fed various amounts of 2,4,5-T were assayed for 2,4,5-T and 2,4,5-trichlorophenol employing an extraction with 2% ammonium hydroxide in methanol. Other portions of these samples were subjected to hydrolysis with both 6N KOH and 6N H₂SO₄ before extraction to obtain the total residue and to compare the effects of both acid and alkali on the residues obtained. Still other portions of these samples were extracted with 2% ammonium hydroxide-methanol mixture and the extract and the extracted solids were hydrolyzed by both 0.5N KOH and 0.5N H₂SO₄. Interesting residue patterns were obtained.

23. DETECTION OF MAMMALIAN EXPOSURE TO CARBAMATE INSECTICIDES. Hazel C. Sullivan, T. Shafik, Perrine Primate Laboratory, Environmental Protection Agency, P. O. Box 490, Perrine, Florida 33157.

A method for the determination of low level exposure in mammals to three carbamate insecticides (Baygon, carbaryl, carbofuran) will be described. The procedure is based on the quantitation of the corresponding phenolic or hydroxylated metabolite in urine. Since in each case, the metabolite to be detected had poor electron capturing properties, the pentafluorobenzyl ether derivative was used to enhance the limits of detection. The urine from male rats fed at levels as low as 10⁻² of the LD₅₀ was analyzed using acid hydrolysis, extraction, derivatization, silica gel cleanup, and electron capture gas chromatography. Data will be presented which establishes the limits of detection and the rate of excretion of the three metabolites used as the index of exposure to these insecticides.

24. DETERMINATION OF METALDEHYDE IN CROP TISSUE. Sami Selim and James N. Seiber, Department of Environmental Toxicology, University of California, Davis, Calif., 95616.

A new method for the determination of the molluscicide metaldehyde in crop tissue has been developed. After extraction with benzene and cleanup on Florisil, metaldehyde was converted to acetaldehyde, which was determined as its 2,4-dinitrophenylhydrazone derivative using alkali-flame ionization gas chromatography. Recoveries of 94 to 102 percent were obtained for artichoke and strawberry samples fortified at 0.1, 1.0, and 10 ppm. The method which offers important advantages over an existing procedure [J. Agr. Food Chem., 12, 249 (1964)], was used in dissipation studies with artichokes and strawberries treated with several formulations. Residues of metaldehyde decreased to below 0.1 ppm within 14 days following application in all cases except with bait-treated strawberries in which more persistent residues were encountered. The results will be discussed in terms of the chemical stability and volatility of metaldehyde.

25. DEVELOPMENT OF METHODS USING 1-FLUORO-2,4-DINITROBENZENE FOR GAS-LIQUID CHROMATOGRAPHY OF METHOMYL. C. E. Mendoza, J. B. Shields, Food Directorate, Health Protection Branch, National Health & Welfare, Ottawa, Canada.

A method using 1-fluoro-2,4-dinitrobenzene (DNFB) was successfully developed for determination of methomyl or Lannate^(R). Methomyl was hydrolyzed by sodium hydroxide (NaOH) immediately before reaction with DNFB. The methylamine derived from methomyl was reacted with DNFB at 80-82°C in a water bath equipped with a shaker. The reaction product dinitrophenylmethylamine (DNPMA) was analyzed by gas-liquid chromatography. The recoveries of methomyl added to rape seed oil at 0.05 to 1 ppm ranged from 80 to 105%, with an average of about 97%. The technique also gave quantitative recoveries of DNPMA from methomyl (anti) isomer and carbaryl standards. An extra product, other than DNPMA, was obtained from Zectran^(R); it is currently under investigation.

26. DIRECT GAS CHROMATOGRAPHIC ANALYSIS OF METHOMYL RESIDUES IN SOIL, SEDIMENT, WATER AND TOBACCO UTILIZING THE FLAME PHOTOMETRIC DETECTOR. Robert G. Reeves and D. W. Woodham, P. O. Box 3296, USDA, APHIS, PPQP, Environmental Quality Laboratory, Brownsville, Texas 78520.

An improved method for the direct gas chromatographic analysis of methomyl in soil, sediment, water and tobacco is described. Methomyl is extracted from soil, sediment and water with dichloromethane and from tobacco with a mixture of 97.5% dichloromethane and 2.5% benzene. The extracts are further purified by elution through a chromatographic column of activated Florisil^R. Following solvent concentration, the samples are injected on the gas-liquid chromatograph (GLC) equipped with a 394 mμ sulfur interference filter. Residues as low as 0.05 ppm may be detected in 50 gram tobacco samples, 150 gram soil or sediment samples or 300 gram water samples. The procedure is simple, reliable and rapid for environmental samples of these and other types.

27. RESIDUES OF DIMETHOATE AND ITS OXYGEN ANALOG ON AND IN CITRUS LEAVES FOLLOWING A HELICOPTER TREATMENT OF THE TREES WITH A DIMETHOATE ULV CONCENTRATE AND HIGH VOLUME SPRAY. Donald W. Woodham, Robert G. Reeves, P. O. Box 3296, USDA, APHIS, PPQP, Environmental Quality Laboratory, Brownsville, Texas 78520., Charles B. Williams, Harry Richardson, Charles A. Bond, P. O. Box 1829, USDA, APHIS, PPQP, Plant Protection District Hdqs. Harlingen, Texas 78550.

Residues of dimethoate and its oxygen analog on and in citrus leaves were investigated by a gas chromatographic-flame photometric detection (GC-FPD) procedure following

treatment of the trees with (1) a dimethoate ultra low volume (ULV) concentrate and (2) a dimethoate high volume (HV) spray, both applied at a rate of 1.0 lb./A. Residues detected one day following treatment indicated a uniform deposition of the insecticide for both types of application. This was true for all segments of the tree, including the bottom and center sections. The ULV treatment produced higher initial residues than the high volume spray, probably due to excessive runoff of the aqueous HV spray solution from the waxy leaf surface. After 14 days weathering, residues on leaves from the ULV treated trees were slightly higher than those on leaves from the HV sprayed trees.

28. ESTERS OF SULFONYL CHLORIDES AS DERIVATIVES FOR THE GAS CHROMATOGRAPHIC ANALYSIS OF CARBAMATE PESTICIDES: H. A. Moye, Pesticide Research Laboratory, Department of Food Science, University of Florida, Gainesville, Florida, 32601.

Aromatic sulfonyl chlorides, under appropriate conditions, may be reacted with the phenolic portions of hydrolyzed carbamate pesticides to produce esters which are easily gas chromatographed. Sulfonyl chlorides were chosen that would produce esters which would be highly sensitive to the electron capture detector. Those reagents studied were the pentafluoro-, p-bromo-, 2,5-dichloro-, and 3,4-dichlorobenzene sulfonylchlorides. Reaction conditions for the derivatizations were optimized. The hydrolytic and thermal stability of the esters were determined and compared to other derivatives of choice. The optimum gas chromatographic conditions were determined. A procedure for the analysis of carbofuran on lettuce was developed, which was sensitive to 0.01 ppm.

29. RESPONSE OF THE ELECTROLYTIC CONDUCTIVITY DETECTOR IN THE OXIDATIVE MODE TO HERBICIDES AND ITS USE IN RESIDUE ANALYSIS IN SOIL AND WATER BY GAS CHROMATOGRAPHY. R. Purkayastha, Chem. & Biol. Res. Institute, Canada Agriculture, Research Branch, Ottawa, Ontario K1A-0C6. CANADA.

Eighteen herbicides representing four major groups, s-triazines, phenoxy acids, carbamates, and substituted ureas, have been studied by gas chromatography (GC) and their responses measured with a Coulson electrolytic conductivity detector operated in the oxidative mode wherein pyrolysis of GC peaks occur in the presence of excess oxygen at 750-800°C. The 50% full scale deflection (f.s.d.) values obtained with a 5ft x 1/4 inch 3% SE-30 column at 185°C are in the range of 14 - 150 ng. Fifteen of the compounds show 50% f.s.d. values < 60 ng. The following list of compounds is arranged in order of decreasing sensitivity (i.e. increasing 50% f.s.d. values): diallate, triallate, dicamba methyl ester, silvex methyl ester, 2,4-DB methyl ester, vernolate, eptam, CDEC, simazine, 2,4,5-T methyl ester, 2,4-D methyl ester, molinate, atrazine, propazine, prometryne, ametryne, linuron and atratone. The sensitivity of the detector is found to be sufficient to allow for development of residue methods for soil and water samples. Satisfactory results have been obtained in the simultaneous analysis of atrazine and linuron in soil, linuron in water, and 2,4-D in soil and water. The recovery from fortified samples has been fair to excellent in the 0.1-1.0 ppm range.

30. THEORY OF CONTROLLED RELEASE--DIFFUSION.

R. L. Collins and S. Doglia, Physics Department, The University of Texas at Austin, Austin, Texas 78712.

Diffusion is the oldest of the methodologies for controlled release of pesticides. The solution to Fick's second law of diffusion, $D\nabla^2\theta - \dot{\theta} = 0$, can be obtained using error functions or by separation of variables. The error function solutions lead to an initial $t^{-1/2}$ rate law. If the active substance decays with first-order rate constant λ , the present concentration is $C = \int_0^t R(t')e^{-\lambda(t-t')}dt'$ where $R(t')$ is the rate of release. The error function solution cannot be integrated, but the solutions based on separation of variables can. This leads to

$$C = K \sum_{n=1}^{\infty} \frac{e^{-n^2 t \ln 2 / \tau} - e^{-t \ln 2 / \tau_0}}{\tau / \tau_0 - n^2}$$

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K is a constant, τ_0 is the half-life of the active material, and τ is the effective half-life of the diffusion-controlled release package. The effective persistence τ is readily changed by choice of matrix and of geometry. With τ at our disposal, an optimum τ for any needed protection time can be engineered. This permits a constant efficiency of utilization of pesticide, independent time, at 30% or 22% of ideal for wafer or granule geometries.

31. ACARICIDAL ENOL ESTERS OF 2-ARYL-1,3-INDANDIONES, STRUCTURE-ACTIVITY RELATIONSHIPS, J. A. Durden, A. A. Sousa, R/D Department, Union Carbide Corporation, South Charleston, West Virginia 25303; J. F. Stephen, Ciba-Geigy, Ardsley, New York. 10502.

Certain of the subject compounds have demonstrated a high degree of miticidal, particularly ovicidal, activity in laboratory and field tests. Extensive structure-activity studies have revealed rather specific substitution requirements on the 2-aryl ring for miticidal activity. Enol esters may elicit a marked miticidal response in cases where the parent indandione demonstrates minimal activity, depending on the nature of the substitution on the 2-aryl ring. For the more effective indandiones the corresponding enol esters are generally equiactive with the parent at the adult LD₅₀ level but possess markedly steeper dose-response curves. The enol esters are usually more effective ovicides than are the indandiones. The enol ester provides enhanced transport properties in the biological system. These studies suggest that generation of the parent indandione in situ is necessary for a toxic response. The lower alkanoyl esters possess fumigant properties not found with the higher alkanoyl derivatives of the parent indandiones. These compounds are characterized by a low degree of mammalian toxicity. One of these esters, UC 41305 (3-pivaloyloxy-2-(2',4',6'-trimethylphenyl)indone), is scheduled for further development in 1973.

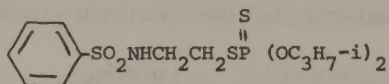
TUESDAY AFTERNOON

32. SELECTIVE HERBICIDAL ACTIVITY. R.L. Wain. Wye College, University of London, Wye, Ashford, Kent, England.

Factors which can operate in selective herbicidal action will be examined. These include the physical properties of the herbicide and its method of formulation. In some cases selectivity is due to biochemical modification of the molecule occurring to different extents in weed and crop. On the one hand, the applied chemical may be harmless per se to the crop but readily converted to a herbicidal substance in susceptible species. Alternatively, a herbicide may become detoxicated to a greater extent in the crop plant than in the susceptible weed. Studies on chemical structure in relation to herbicidal activity within a closely defined series of compounds often provides useful information on mode of action and a lead to the synthesis of more active analogues. Such investigations may also reveal that electronic or steric influences exerted by substituent groupings within the molecule are important in determining the herbicidal activity shown. Although most new herbicides arise from routine screening procedures, knowledge of the above factors can sometimes indicate a more rational approach. An account will be given of investigations of this kind, carried out at Wye College, which have led to the discovery of selective herbicides, some of which are widely used in agriculture. INTERNATIONAL PESTICIDE CHEMISTRY RESEARCH AWARD ADDRESS.

SYMPOSIUM ON NEW ASPECTS OF ORGANOPHOSPHORUS PESTICIDES - (Concluded) - T. R. Fukuto, Presiding

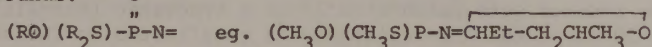
33. BENSULFIDE, S-(O,O-DIISOPROPYL PHOSPHORODITHIOATE) OF N-(2-MERCAPTO ETHYL) BENZENE SULFONAMIDE. Llewellyn W. Fancher, Raymond A. Simone, Eugene G. Teach, Western Research Center, Stauffer Chemical Company, 1200 S. 47th Street, Richmond, California 94804.



Bensulide or Betasan is a selective pre-emergence herbicide which controls certain annual grasses and broadleaf weeds. It is used commercially for weed control in a number of crops including most of the cucurbits, lettuce and cotton. It is also used extensively to control crab grass in home lawns and to control annual bluegrass or poa annua in golf greens. The synthesis of Bensulide will be discussed as well as the effect of substitution at various sites in the molecule.

34. THIOPHOSPHORYLIMINO OXAZOLIDINES, THIAZOLIDINES, AND RELATED COMPOUNDS AS INSECTICIDES, Edmund J. Gaughan, Julius J. Menn, Stauffer Chemical Co., Richmond, California, and Mountain View, California. 94040.

3-(Thiophosphorylimino-) oxazolidines, thiazolidines, thiazolines, and perhydro-oxazines have proven to exhibit a high degree of insecticidal and systemic acaricidal activity. In all of these series, optimal biological activity was realized with the thiophosphorylimino compounds:



The methyl esters of the above structure were usually more active than other homologs. The corresponding thiono isomers were of significantly lower activity. Structure activity studies also revealed that the bulk of the alkyl substituent on the heterocycle ring nitrogen atom was also critical for insecticidal action with smaller substituents showing higher activity. The saturated thiazolidines proved to be more active than the unsaturated thiazoline analogs. With the oxygen containing heterocycles, the five membered oxazolidines were more toxic to insect species than the six membered perhydrooxazines. The influence of geometrical and optical isomerism in the oxazolidine ring on insecticidal activity and mammalian toxicity was also investigated.

35. METABOLISM, BIOCHEMICAL AND BIOLOGICAL ASPECTS OF NEMACUR AND RELATED PHOSPHORAMIDATE COMPOUNDS. T. Bill Waggoner, Abdullah M. Khasawinah, Chemagro Division of Baychem Corporation, Box 4913, Kansas City, Mo. 64120.

The metabolism of Nemacur [Ethyl 4-(methylthio)-*m*-tolyl isopropylphosphoramidate], a new nematocide now being developed, has been reported by Waggoner [*J. Agr. Food Chem* 20, 157 (1972)] for tomato, bean and peanut plants, and the major metabolites were identified as the corresponding sulfoxide and sulfone. In this paper, further results will be presented for Nemacur in various plants following soil treatment and in animals. The major metabolic route in soil, plants and animals is oxidation, hydrolysis and subsequent conjugation of the phenols. Metabolic data from tests with ethoxy-¹⁴C and methylthio-³H labels and field residue data will be discussed for tobacco, legumes, root crops and other vegetables. Excretion of radioactivity from ethoxy-¹⁴C labeled Nemacur in rats is quantitative within 48 hours after an oral dose. *In vitro* studies with rat liver microsomes have shown complete metabolism of Nemacur (ethoxy-¹⁴C and methylthio-³H labels) within 10 minutes: an *N*-dealkylated metabolite was identified. Toxicity data, chemistry and biological activity of several structurally related Nemacur compounds will be discussed.

36. SOME RECENT ADVANCES IN THE STUDY OF ORGANOPHOSPHATE METABOLISM. Robert M. Hollingworth, Department of Entomology, Purdue University, W. Lafayette, Indiana 47907.

At present three distinct types of enzymic reactions are known which degrade insecticidal organophosphorus triesters at the acid anhydride bond to yield innocuous diesters e.g. the conversion of paraoxon (diethyl *p*-nitrophenyl phosphate) to diethyl phosphoric acid. These mechanisms are: -

- (1) Phosphotriesterases (A-esterases).
- (2) Microsomal mixed function oxidases.
- (3) Glutathione transferases.

Aspects of current research on each of these mechanisms will be discussed as they relate to the metabolism of the phosphate (P=O) esters. Emphasis will be placed on the nature and basis of substrate specificity of these enzymes, their comparative enzymology in vertebrates and invertebrates and their potential significance in selective toxicity.

PEST

WEDNESDAY MORNING - SYMPOSIUM ON BIOCHEMISTRY OF INSECT RESISTANCE - F. W. Plapp, Jr., Presiding

37. BIOCHEMISTRY OF INSECT RESISTANCE TO DDT. Moises Agosin, University of Georgia, Athens, Georgia 30601.

Resistance to DDT is brought about by DDT-ase, microsomal monooxygenases or both. The molecular aspects of DDT-ase are reported by Dinamarca *et al.* [Arch. Biochem. Biophys., 147, 374 (1972)]. Microsomal-dependent DDT-resistance is often correlated with cross-resistance to unrelated insecticides. The latter are capable of binding to site I of cytochrome P-450, the hydroxylation enzyme. Neither the levels of cytochrome P-450 nor its reductase are rate limiting. Cytochrome P-450 is induced by DDT, phenobarbital, and naphthalene. An inducer binds to site I and can stimulate the hydroxylation of an unrelated substrate only when the latter is capable of displacing it from site I. Otherwise, the inducer acts paradoxically as a synergist (Morello *et al.*, unpublished). Cytochrome P-450-dependent DDT-resistance is complicated by the existence of at least two forms of hemoprotein, control P-450 and induced P-446. Induced P-446 metabolizes substrates which have greater affinity for its site I at a higher rate. Furthermore, cytochrome P-450 and P-446 show type I spectral changes only when they are reduced or in the presence of ETNC. Cytochrome P-446 shows large type I changes at low substrate concentrations, while cytochrome P-450 requires high concentrations for similar changes (Capdevila *et al.*, unpublished). Allosteric modulation will be discussed in this respect.

38. GENETIC STUDIES ON FACTORS AFFECTING CROSS-RESISTANCE SPECTRA IN INSECTICIDE-RESISTANT INSECTS. Roman M. Sawicki, Rothamsted Experimental Station, Harpenden, Hertfordshire, England.

The isolation and identification of individual factors of resistance in strains of insects with polyfactorial resistance by means of genetic techniques, has led to a better understanding of some important problems of resistance.

(i) Strong resistance, especially to organophosphate insecticides often occurs in strains of house flies in which individual mechanisms of resistance to these compounds are weak. Re-synthesis of multi-resistance from strains with single mechanisms restores strong resistance. This indicates that strong resistance results from interactions between weak mechanisms. Several such interactions are now known and are discussed.

(ii) House flies often develop resistance to new organophosphate insecticides without losing previously acquired resistance to other organophosphates. When dimethoate was used to control resistance to parathion and diazinon, house flies developed resistance to this insecticide without apparently losing previously acquired resistance. Genetic analysis showed that the resistance to diazinon and parathion in dimethoate resistant house flies was caused only by dimethoate resistance mechanisms that developed at the expense of previously present mechanisms. The implications of this finding are discussed.

39. BIOCHEMISTRY OF INSECT RESISTANCE. NON-MICROSOMAL MECHANISMS. Naoki Motoyama, W.C. Dauterman, Department of Entomology, North Carolina State University, Raleigh, North Carolina 27607.

Non-microsomal mechanisms of insect resistance to organophosphorus insecticides include non-microsomal metabolism, modification of target enzymes (cholinesterases), and difference in rates of penetration. Carboxyesterases of malathion resistant strains of insects and mites have been reported with regard to purification, kinetics, and specificity. The contribution of glutathione transferases to organophosphate resistance mechanisms has recently been suggested. The enzymes catalyze conjugation reactions of alkyl or aryl moieties of organophosphorus insecticides with glutathione. The transferases are examined in relation to detoxification enzymes previously described as phosphatases. The possible role of non-specific esterases detected with α - or β -naphthyl acetate in resistance mechanisms is also discussed. The existence of cholinesterases insensitive to organophosphate and carbamate inhibitors has been reported with insect, mite, and tick species. The enzyme was partially purified from resistant mites and the abnormal properties investigated. Although studies on penetration are relatively few, some cases have been reported in which difference in rates of penetration was a factor in organophosphate resistance.

40. INSECTICIDE RESISTANCE FACTORS IN LEPIDOPTEROUS LARVAE. Robert I. Krieger, Department of Environmental Toxicology, University of California, Davis, Ca 95616.

Among insects of the order Lepidoptera are some extremely important pest species such as the bollworm Heliothis zea, tobacco budworm H. virescens, cabbage looper Trichoplusia ni, and cotton leafworm Spodoptera litura. Certain strains of caterpillars of these and other species are known to be resistant to organo-chlorine, organophosphate and carbamate insecticides. Resistance has been associated with slow rates of absorption and rapid excretion of water-soluble metabolites. Resistant strains also have been found to have higher levels of NADPH-dependent oxidative enzymes than susceptible strains. These oxidase systems can be inhibited by insecticide synergists such as methylenedioxyphenyl compounds and by carbon monoxide indicating an important role for microsomal cytochrome P-450 in oxidation. The biochemical properties of caterpillar oxidase systems will be discussed with particular attention being given to differences in activity associated with species, strain, tissues and larval development.

WEDNESDAY AFTERNOON - SECTION A - SYMPOSIUM ON BIOCHEMISTRY OF INSECT RESISTANCE - (Concluded) - F. W. Plapp, Jr., Presiding

41. THIRD GENERATION PESTICIDES: THE POTENTIAL FOR THE DEVELOPMENT OF RESISTANCE BY INSECTS. S. Bradleigh Vinson, Dept. of Entomology, Texas A&M University, College Station, Texas 77843.

Certain strains of insects showing differences in response to insecticides also show differences in response to insect juvenile hormone analogs. Insect juvenile hormones and their analogs have been shown to be susceptible to both hydrolysis and oxidation. Several juvenile hormone analogs have shown the ability to block as well as induce microsomal enzyme activity. This information as well as the juvenile hormone mimicking effect of certain synergists suggests that juvenile hormones and their analogs are metabolized by the same xenobiotic oxidative system as for the present pesticides. Thus, certain insects may already possess varying degrees of cross-resistance to these potential control agents. Experience with present pesticides suggests that through selection with these third generation pesticides, high levels of resistance may rapidly develop.

42. MICROSOMAL CYTOCHROME P-450: CHARACTERIZATION AND POSSIBLE ROLE IN INSECTICIDE RESISTANCE IN MUSCA DOMESTICA. Ernest Hodgson, L. G. Tate, A. P. Kulkarni, Dept. of Entomology, North Carolina State University, Raleigh, North Carolina, 27607, and F. W. Plapp, Jr., Dept. of Entomology, Texas A&M University, College Station, Texas 77840.

Cytochrome P-450 plays a central role in oxidative metabolism of xenobiotics in insects and mammals. Recent studies have shown increased levels of cytochrome P-450 in insecticide resistance strains of the housefly, Musca domestica L. and that in resistant strains this cytochrome is qualitatively as well as quantitatively different from that in susceptible strains. Cytochrome P-450 has been characterized by examination of the following difference spectra: Carbon monoxide; type I substrate; type II substrate; type III substrate (a new type of substrate difference spectrum obtained from both insects and mammals with methylenedioxyphenyl compounds and recently characterized in this laboratory); n-octylamine. It is probable that more than one cytochrome P-450 occurs in the housefly and that these resemble the cytochromes P-450 from normal and induced mammals. Current work centers around the correlation of P-450 types with the genetics of insecticide resistance and on the structure-function relationships involved in the formation of different types of substrate difference spectra particularly with pesticide substrates.

43. THE INDUCTION OF DETOXYFYING ENZYMES IN INSECTS. L. C. Terriere, Depts. of Agricultural Chemistry and Entomology, Oregon State University, Corvallis, Oregon 97331.

Several microsomal oxidases as well as DDT-dehydrochlorinase show increased activity in houseflies treated with inducing chemicals. The increases occur after either contact or stomach exposure to the chemical and may range as high as 25-fold after the ingestion of some types of inducers. Although only a few compounds have been tested to date, it is clear that the effect is common to several types as is the case with vertebrate animals. Active compounds include DDT, the cyclodiene

insecticides, certain juvenile hormone analogs, and phenobarbital, a barbiturate. Some structural specificity is involved as is shown by the potency of endrin compared to dieldrin and by the specificity of action seen in the JH analogs, one compound inducing DDT-dehydrochlorinase and inhibiting the microsomal oxidases while another has no effect on either enzyme system. With some inducers such as dieldrin and heptachlor there is a greater net increase in enzyme action with resistant insects than with susceptible insects. The phenomenon appears to involve the regulation of enzyme activity and, in the case of the insect hormones, may be related in some way to their morphogenetic effects.

44. EVIDENCE FOR A KEY ROLE OF d-AMINOLEVULINIC ACID SYNTHETASE IN INSECTICIDE RESISTANCE. Frederick W. Plapp, Jr., Dept. of Entomology, Texas A&M University, College Station, Texas 77843.

Resistance to insecticides frequently involves quantitative and qualitative changes in the NADPH-dependent microsomal oxidase system which degrades insecticides and other xenobiotics. Certain chemicals which react with cytochrom P-450, the hydroxylating enzyme, are competitive inhibitors of the oxidase system and act as synergists. Other synergists which appear to block microsomal resistance mechanisms do not inhibit cytochrome P-450 either *in vitro* or *in vivo*. Some of these have been reported to induce d-aminolevulinic acid (ALA) synthetase, the rate-controlling enzyme for the heme biosynthetic pathway in both animals and plants. Chemicals which affect this enzyme influence the production of heme which is necessary for the synthesis of cytochrome P-450 as well as hemoglobins, chlorophylls, etc. Data are presented showing that many compounds which affect ALA synthetase are powerful insecticide synergists or antagonists. The same materials are frequently inhibitors of growth and photosynthesis in algae.

WEDNESDAY AFTERNOON - SECTION B - GENERAL - R. D. Moss, Presiding

45. EFFECT OF STRUCTURE ON THE REACTIVITY OF DIMETHYL SUBSTITUTED-PHENYL PHOSPHATES WITH NUCLEOPHILIC REAGENTS. D. Wendell Osborne, Violete Stevens, Ag-Organics Department, Dow Chemical Co., Midland, Michigan 48640.

Nucleophilic reagents react with dimethyl substituted-phenyl phosphates both by displacement on phosphorus, which results in phosphorylation, and on carbon, which results in methylation. Phosphorylation has been associated with the insecticidal properties of these compounds while alkylation has been associated with their metabolic decomposition. Rate constants for the two modes of reaction were determined for a series of dimethyl substituted-phenyl phosphates using pyrrolidine as the nucleophilic reagent. The rates of both reactions increased with increasingly electron withdrawing substituents, but the phosphorylation reaction was appreciably more sensitive than the alkylation reaction. Compounds with one ortho-chlorine substituent behaved normally and could be integrated with the ortho-unsubstituted compounds by assigning it a pseudo-sigma substituent constant of +0.44. Compounds with two ortho-chlorine substituents behaved abnormally. The housefly and mouse toxicities as well as the ability to inhibit acetylcholinesterase all generally increased with increasingly electron withdrawing substituents in this series. No clear correlation, however, could be made between the ratio of the mouse and housefly toxicities and substituent constants.

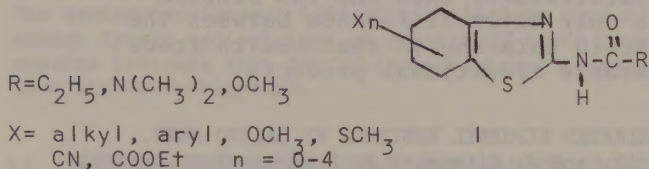
46. SYNTHESIS AND BIOLOGICAL ACTIVITY OF A NEW CLASS OF OXIME CARBAMATES. T. A. Magee, Diamond Shamrock Corporation, P. O. Box 348, Painesville, Ohio 44077, L. E. Limpel, Boyce Thompson Institute for Plant Research, Inc. Yonkers, New York 10701.

A series of ketoxime carbamates, some of which show excellent insecticidal and acaricidal activity, will be discussed. One of the compounds, extensively field tested under the code number DS-15647 (ENT 27851), has exhibited outstanding activity as a soil applied systemic insecticide on cotton, potatoes, sorghum and sugar beets. Use of DS-15647 as a seed treat-

ment has given excellent early season control of insects on cotton. The insecticidal structure-activity relationships will be compared and contrasted with a series of trisubstituted acetaldehyde oxime carbamates described in the literature.

47. TETRAHYDROBENZOTHAIAZOLE HERBICIDES. - I John E. Engelhart, M&T Chemicals Inc., Research Laboratory, Woodbridge Road and Randolph Avenue, P.O. Box 1104, Rahway, New Jersey 07065.

An investigation of the tetrahydrobenzothiazole ring system has uncovered a new class of very active herbicides. These herbicides are all derivatives of a series of 2-amino-4,5,6,7-tetrahydrobenzothiazoles as exemplified by structure I.

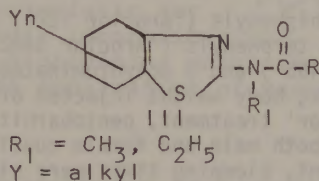
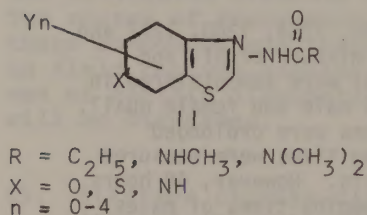


Methods of synthesis of compounds of this class will be discussed starting with various alkyl, alkoxy, alkylthio and cyano substituted cyclohexanones. Amide and urea derivatives of the amino function will be particularly stressed because of their inherent high level of activity. Structure-activity relationships will also be described, outlining the need for ring substituents at the 5- and 7- positions for optimal activity. The herbicidal activity in this series will be illustrated by the results of extensive greenhouse tests with a series of broadleaf and grassy crops and weeds.

Post-emergence herbicide activity for several of these compounds is apparent at the 0.1 lb/acre dosage rate. The general lack of significant pre-emergence activity in this series will also be discussed.

48. TETRAHYDROBENZOTHAIAZOLE HERBICIDES - II, John E. Engelhart, M&T Chemicals Inc., Research Laboratory, Woodbridge Road and Randolph Avenue, P. O. Box 1104, Rahway, New Jersey 07065.

A further description of our work in the tetrahydrobenzothiazole series will illustrate the effect of heteroatom substitution in the 6-membered ring and the effect of alkyl substitution on the amino group of the 2-aminotetrahydrobenzothiazoles. These systems can be exemplified by the following structures, II and III.



Synthesis of tetrahydrobenzothiazoles containing oxygen, sulfur or nitrogen in the 6-membered ring will be described utilizing various tetrahydropyrones, tetrahydrothiapyrones and diacetoneamines. In addition, conversion of 2-aminotetrahydrobenzothiazoles to the corresponding N-methyl and N-ethyl analogs followed by derivatization to amides and ureas will be outlined.

The effects of such molecular alterations on herbicide properties will be shown by the results of extensive greenhouse tests on broadleaf and grassy crops and weeds. A general summary of herbicide activity in the tetrahydrobenzothiazoles will be presented at the conclusion of this talk.

49. PCB CONTENT OF SELECTED HEALTH FOODS. W. B. Wheeler Pesticide Research Laboratory, Food Science Department, J. A. Koburger and H. Appledorf, Food Science Department, University of Florida, Gainesville, Fla. 32601.

Twenty-five paired food samples each composed of a "health" food and a comparable "traditional" food were analyzed for pesticide and PCB content; aerobic plate count and coliforms; and proximate composition. Analyses for pesticides and PCB's revealed the presence of PCB's in seven health food samples and three traditional foods while pesticides were not detected within the limits of the method employed. Microbiological data showed the presence of coliforms in three of 25 traditional foods and six of 25 health foods. Some aerobic plate counts were excessively high in both groups of foods. Proximate compositional data were similar, particularly when the two samples were from the same processor. The only major difference between the groups of foods was price. Cost ratio data showed that health foods averaged higher in cost than comparable traditional products.

50. RETENTION AND EXCRETION OF POLYCHLORINATED BIPHENYL RESIDUES BY LAYING HENS.

G. F. Fries, R. J. Lillie, H. C. Cecil, and J. Bitman, U.S. Department of Agriculture, ARS, Agricultural Research Center, Beltsville, Md. 20705.

Polychlorinated biphenyl (PCB) mixtures ranging from 21 to 68% chlorine were individually fed to groups of hens. All PCBs were fed at 20 mg/kg in the diet and three (42, 48, and 54% chlorine) were also fed at 2 mg/kg. Residues were determined in composite egg samples and excreta samples weekly during the 9-week feeding period and the 7-week period after feeding stopped. Individual egg and body fat samples from five hens of each group were obtained at 9 and 16 weeks. The residues in excreta qualitatively resembled the mixtures fed, but only 5 to 10% of intake was excreted from each mixture. In eggs and body fat the less chlorinated components (tetra or less) were either absent or present in reduced relative concentrations. Thus, total PCB residue found in eggs and body fat increased with increasing chlorination of the PCB mixture fed. The 42 and 68% mixtures were exceptions. The 42% mixture caused lower egg production reducing elimination by this route and causing enhanced residue levels in eggs and body fat. With the 68% mixture, the more highly chlorinated components (octa and greater) were eliminated at higher levels in the eggs and retained at lower levels in the body fat. In general, the greatest residue levels relative to intake occur with the components of intermediate degrees of chlorination. It is hypothesized that the less chlorinated components are metabolized, and that the more chlorinated components do not readily cross biological membranes.

51. EFFECTS OF A SINGLE ORAL INJECTION OF POLYCHLORINATED BIPHENYLS AND TERPHENYLS AND A POLYBROMINATED BIPHENYL ON PENTOBARBITAL SLEEPING TIMES OF JAPANESE QUAIL. Helene C. Cecil, Susan J. Harris and Joel Bitman, Biochemistry Laboratory, Agricultural Research Service, Animal Physiology and Genetics Institute, U. S. Department of Agriculture, Beltsville, Maryland 20705.

Polychlorinated biphenyls ('Aroclor 1221, 1232, 1242, 1248, 1254, 1260, 1262 and 1268') polychlorinated terphenyls ('Aroclor 5442 and 5460'), a mixture of bi- and terphenyls ('Aroclor 4465') and a polybrominated biphenyl (BP-6) were administered in a single dose, 100 mg/kg body weight injected orally, to mature male and female quail. Two hours after 'Aroclor' treatment, pentobarbital sleeping times were prolonged (2 to 3 x control) in both male and female quail. When sleeping times were measured 24 hours after treatment, sleeping times were similar to controls. However, 48 hours after treatment with 'Aroclors 1248 through 5460' and BP-6, sleeping times of males were only 1/2 that of the controls while males treated with PCB 1221, 1232 and 1242 were similar to controls. Greater effects were observed in male than female quail. Although very few birds died from the toxicity of the 'Aroclors' and BP-6, mortality was greatly increased during anesthesia when the pentobarbital was administered 2 hours after dosing.

52. RETENTION AND EXCRETION OF POLYBROMINATED BIPHENYLS BY HENS AND COWS. G. F. Fries, L. W. Smith, H. C. Cecil, J. Bitman and R. J. Lillie, U.S. Department of Agriculture ARS, Agricultural Research Center, Beltsville, Md. 20705.

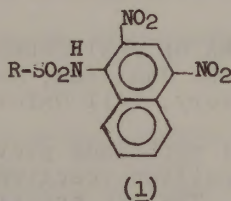
A commercial polybrominated biphenyl mixture containing 75% bromine was studied in two animal species to establish the major parameters of residue retention and excretion.

The mixture contained two major and several minor components. The major components were identified by mass spectrometry as a hexabromobiphenyl and a heptabromobiphenyl. Routine methods for chlorinated hydrocarbons were adequate for analyses except that a column that could withstand higher temperatures was required for electron capture gas-liquid chromatography. The polybrominated biphenyl was fed to 30 hens at 20 mg/kg in the diet for 9 weeks. Composite egg samples and excreta samples were obtained from the hens weekly during the 9-week feeding period and 7-week period after feeding stopped. Five individual egg samples and body fat samples were analyzed at 9 and 16 weeks. Chicken excreta collected during the 9-week feeding period was dried and incorporated in the ration of a lactating cow for 7 weeks. Average concentration in the ration was about 0.5 mg/kg. Milk samples were taken at 5-day intervals during the feeding period and during the 7-week period following feeding. The hexabromobiphenyl, accounting for 70% of the electron capture response in the original material, was retained and excreted in both species at rates typical of chlorinated hydrocarbons resistant to degradation. The heptabromobiphenyl (20% of the electron capture response) was excreted at relatively higher levels and retained at relatively lower levels than the hexabromobiphenyl. The results indicate that the hexabromobiphenyl crossed biological membranes less readily than heptabromobiphenyl.

53. POLYNITRO-SUBSTITUTED ARYLSULFON-1-NAPHTHALIDES AS CHEMOSTERILANTS FOR HOUSE FLIES

Albert B. DeMilo and Alexej B. Bořkovec, Agricultural Environmental Quality Institute, USDA, Agricultural Research Service, ARC, Beltsville, Maryland 20705.

A number of alkyl-, aryl-, and heteroarylsulfon-2,4-dinitro-1-naphthalide derivatives (1) have been synthesized and many were found to be highly effective as chemosterilants for the house fly, *Musca domestica* L., when administered as dietary additives in screening tests.

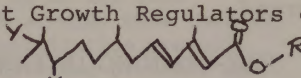


The syntheses, structure-activity relationships, and biological effects of these compounds will be discussed in detail.

54. ELECTRON INDUCED FRAGMENTATION OF CONJUGATED DIENOATE ESTERS.

L. L. Dunham, J. W. Young, C. A. Henrick, Zoecon Research, 975 California Avenue, Palo Alto, CA 94304.

Mass spectral fragmentation characteristics of Insect Growth Regulators of the conjugated dienoate ester series are discussed. The routes of fragmentation are characteristic for these compounds and thereby aid in their identification^x in field residue samples, as well as elucidating metabolic pathways. The use of certain diagnostic ions for the application of mass chromatography will be discussed.



55. ELECTRON CAPTURE-GAS LIQUID CHROMATOGRAPHY OF 2,6-DICHLORO-4-NITRO-ANILINE AND ITS METABOLITE IN TISSUES AND EXCRETA. Robert F. Moseman, EPA-Chamblee Toxicology Laboratory, 4770 Buford Highway, Chamblee, Georgia 30341.

Electron capture-gas liquid chromatographic methods of determination have been developed for the fungicide, Botran (R) (2, 6-dichloro-4-nitro-aniline) and its major metabolite, 3,5-dichloro-4-aminophenol. For the parent compound, extraction, purification and gas chromatography is similar to the scheme used for the determination of chlorinated hydrocarbon insecticide residues. The analysis for the major metabolite, 3,5-dichloro-4-aminophenol requires a hydrolysis step and derivatization with chloroacetic anhydride. The resulting O-chloroacetyl derivative chromato-

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graphs well with a minimum detectable amount of about 5 picograms. Tissues, urine and feces of rats dosed with Botran (R) have been analyzed using these techniques. Recoveries of 90% or better have been obtained for samples fortified with the parent compound and its metabolite.

THURSDAY MORNING - SYMPOSIUM ON MASS SPECTROMETRY AND NMR OF PESTICIDES -
F. J. Biros, Presiding

56. CHARACTERIZATION OF CHLORINATED HYDROCARBON PESTICIDE METABOLITES OF THE DDT AND POLYCYCLODIENE TYPES BY ELECTRON IMPACT AND CHEMICAL IONIZATION MASS SPECTROMETRY.

J. D. McKinney and E. O. Oswald, National Institute of Environmental Health Sciences, National Institutes of Health, Public Health Service and Department of Health, Education and Welfare, P. O. Box 12233, Research Triangle Park, N. C. 27709.

Electron impact (EI) and chemical ionization (CI) mass spectra of the major biological oxidative and synthetic metabolites of aldrin and dieldrin have been obtained. In addition, both CI and EI spectra for essentially all of the known and proposed metabolites of DDT including the aldehyde precursor of DDA and a chemical conjugate of kelthane were examined. The spectra which will be discussed were selected for particular peculiarities and differences which illustrate the advantages of using EI and CI mass spectrometry for characterization of these compounds as potential environmental agents. Other spectra were chosen to demonstrate the utilization of the mass spectrometer as a model system for predicting the susceptibility of certain compounds to rearrange under thermal and photolytic conditions. Computerized processing of the data has enabled the use of these mass spectral systems for rapidly determining the nature of products derived from investigation of model chemical reactions yielding complex mixtures.

57. THE ION KINETIC ENERGY SPECTRA OF PESTICIDES AND RELATED COMPOUNDS.

S. Safe, O. Hutzinger and W. D. Jamieson, National Research Council of Canada, Atlantic Regional Laboratory, 1411 Oxford Street, Halifax, N. S., Canada.

The ion kinetic energy (IKE) technique provides a sensitive method for studying unimolecular decomposition reactions in the first field-free region of the mass spectrometer. The IKE spectra of isomeric DDT-like compounds indicated that the o,p' and p,p' DDD and DDT isomers could be distinguished due to a substituent effect whereas the o,p' and p,p' DDE isomers exhibited complete chlorine atom equilibration. Some possible analytical applications of these results will be discussed. The IKE spectra of the isomeric polychlorinated biphenyls, chloroanilines and chlorophenols gave the following results; 1) each isomer gave a useful fingerprint spectrum which distinguished the compound from the other positional isomers and 2) new ionic decomposition reactions were observed.

58. SELF-TRAINING INTERPRETIVE AND RETRIEVAL SYSTEM FOR MASS SPECTRA.

Kain-Sze Kwok, Rengachari Venkataraghavan, Gail Pesyna, Ikuo Sakai, J. W. Serum, Akira Tatematsu, R. G. Werth, F. W. McLafferty, Department of Chemistry, Cornell University, Ithaca, New York 14850.

A self-training system is described for computer interpretation of mass spectra which utilizes directly data of all available reference spectra, and does not require prior spectra/structure correlations of these data either by human or computer effort. The computer selects different classes of data known to have high structural significance, such as characteristic ions, series of ions, and masses of neutrals lost, from the unknown mass spectrum, and matches these against the corresponding data of all the reference spectra. The reference compounds of closest match in each data class are examined for common structural features; criteria have been determined so that such features can be identified with approximately 95% reliability. The file of reference data is of key importance in the usefulness of STIRS. Over 20,000 mass spectra have been collected from a variety of sources and their reliability checked by both human and computer review. Each compound has been coded in Wiswesser Line Notation; utilization of these for computer identification of structural features will be discussed.

59. POSITIVE AND NEGATIVE CHEMICAL IONIZATION MASS SPECTRA OF POLYCHLORINATED PESTICIDES. Ralph C. Dougherty, J. David Roberts, F. J. Biros; Department of Chemistry, Florida State University, Tallahassee, Florida 32306; Perrine Primate Lab., U.S. Environmental Protection Agency, Perrine, Florida 33157.

Chemical ionization mass spectra are the result of ion molecule reactions of an ionized reagent gas and a much less abundant substrate. The spectra are thus the result of normal ionic chemical reactions that occur in the absence of solvent. CI spectra usually contain fewer types of ions than their electron impact counterparts and they directly provide information about normal ionic reactivity in the absence of a solvent. The major ions in the positive ion (isobutane) CI mass spectra of chlorinated pesticides always include $[M - Cl]^+$. Negative ion CI (NCI) spectra of the same aromatic and polycyclic pesticides always include $[M + Cl]^-$ as a major ion. The chloride ions that are responsible for these attachment reactions result from disassociative electron capture by the parent molecules. For analytical purposes positive CI spectra would appear to be best for polycyclic insecticides while NCI spectra would be most satisfactory for insecticides in the DDT family. The requirements for developing screening procedures for chlorinated pesticides and pesticide metabolites in natural materials based on CI and NCI spectra will be discussed.

The positive CI spectra of DDT related pesticides show a number of ions that must result from aryl migrations and related carbonium ion reactions. We will discuss the significance of these ions and other fragment ions that appear in both the positive and negative CI spectra.

60. MASS SPECTRA OF PESTICIDE PHOTOPRODUCTS. Joseph D. Rosen, College of Agricultural and Environmental Sciences, Department of Food Science, Rutgers University, The State University of New Jersey, New Brunswick, New Jersey 08903.

The use of mass spectroscopy in the determination of photoproducts of pesticides will be discussed.

61. ELECTRON IMPACT MASS SPECTRA OF SEVERAL AROMATIC RING SYSTEMS. A. Curley, R. W. Jennings, V. W. Burse, and E. Villanueva, U.S. Environmental Protection Agency, Chamblee Toxicology Laboratory, 4770 Buford Highway, Chamblee, Georgia 30341.

Several technical polychlorinated hydrocarbon pesticides are synthesized from common precursors. In basic toxicity studies with these compounds, certain effects are observed that may be attributed to contaminants in the technical preparation rather than the parent compound. Mass spectral studies of several chlorinated and non-chlorinated compounds associated with assays for toxic contaminants have revealed fragmentation patterns that might describe interesting ortho-para substituent effects in these aryl systems. The chlorodiphenyl ethers, chlorodibenzodioxins, chlorodibenzofurans, and dibenzofuran have been examined by electron impact on an LKB 9000 combined gas chromatograph-mass spectrometer. These preliminary studies show that the chlorobiphenyl ethers yield furan fragments when ortho-ortho substitution prevails. The chlorodibenzo-p-dioxins lose water when ortho-ortho prime hydrogen prevails and chlorine plus CO when at least one ortho-chlorine is present. The chlorodibenzofurans yield principally a molecular ion minus chlorine and to a lesser extent, when dictated by the position of the chlorine atoms in the aryl system, a cyclopentadienophenylene fused ring skeleton.

These studies might be useful in diagnostic residue studies of contaminants at the submicrogram level.

62. MASS SPECTRAL ANALYSIS OF SOME CARBANILATE HERBICIDES AND THEIR ARYL HYDROXYLATED METABOLITES. Gerald G. Still, Agricultural Research Service, U. S. Department of Agriculture, Metabolism and Radiation Research Laboratory, Fargo, North Dakota 58102.

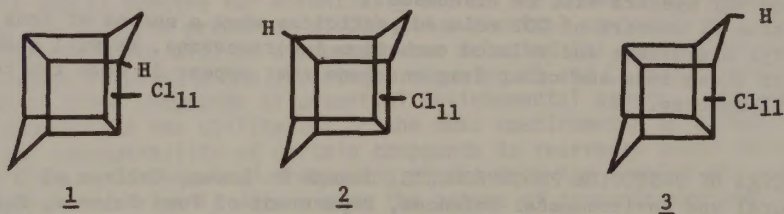
Two widely used herbicides, propham (isopropylcarbanilate) and chlorpropham (isopropyl-3-chlorocarbanilate) have been shown to fragment, under electron-impact, in a manner similar to ethyl N-phenylcarbamate. Both Lewis and Damico have shown that ethyl N-phenylcarbamates, propham and chlorpropham, undergo McLafferty rearrangements to produce fragment ions.

Plants and animals have been shown to hydroxylate the phenyl nucleus of both propham and chlorpropham. Soybean plants metabolize propham to a polar conjugate

whose aglycone is isopropyl-2-hydroxycarbanilate. The O-glucoside of both isopropyl-2-hydroxy-5-chlorocarbanilate and isopropyl-4-hydroxy-3-chlorocarbanilate have been isolated from soybean shoots and shown to be the products of chloropham metabolism. A comparison of the mass spectra of *ortho*, *meta* and *para* hydroxy analogs of isopropyl-3-chloro-hydroxycarbanilate indicates a preferential "ortho" intramolecular rearrangement during the fragmentation of ortho-hydroxy isopropyl-chlorocarbanilates.

63. IDENTIFICATION OF INSECTICIDE PHOTOPRODUCTS BY MASS SPECTROMETRY. Bobby R. Layton and Earl G. Alley, Mississippi State Chemical Laboratory, Mississippi State, Mississippi 39762.

The structures of some photoproducts of the pesticides Mirex and Kepone have been assigned utilizing GC-MS. These photoproducts are derived from the parent compounds by replacement of one or two chlorines by hydrogens. The fragmentation patterns of the monohydro photoproduct of Kepone and chemical inter-relationship between it and the monohydro photoproducts of Mirex permit differentiation between the very similar derivatives of Mirex, 1, 2, and 3, each of which would produce identical mass spectra.



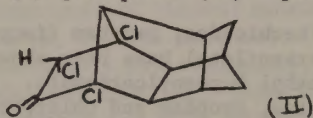
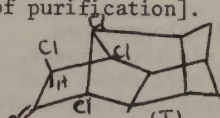
64. THE APPLICATION OF MASS SPECTROMETRY TO THE STUDY OF NITROANILINE DERIVED HERBICIDES. Jack R. Plimmer and Ute I. Klingebiel, USDA, ARS, Agricultural Research Center, Plant Industry Station, Beltsville, Md. 20705.

The photochemistry and metabolism of several dinitroaniline herbicides including N-secondary-butyl-4-tertiary-butyl-2,6-dinitroaniline (A 820) and N³,N³-diethyl-2,4-dinitro-6-trifluoromethyl-m-phenylenediamine (dinitramine) are being investigated. Mass spectrometry has been used as the primary technique for structural investigation since quantities of products are limited. High resolution electron impact mass spectra provide confirmation of fragmentation pathways that are characteristic of substituted anilino, nitro, trifluoromethyl, and other ring substituents. Abundant ions result from charge localization at the nitrogen atom of the aniline group. Formation of substituted benzimidazoles or benzoxazoles is accompanied by the appearance of relatively abundant characteristic ions.

THURSDAY AFTERNOON - SYMPOSIUM ON MASS SPECTROMETRY AND NMR OF PESTICIDES - (Continued) - R. Haque, Presiding

65. ¹H NMR SPECTRA AND STRUCTURE OF A UNIQUE SATURATED TETRACHLOROPENTACYCLODODECANONE DERIVED FROM MONOCHLOROALDRIN AND MONOCHLOROISODRIN. K. Mackenzie, C.H.M. Adams D. J. Cawley, University of Bristol, England.

The hexachlorocyclodiene pesticides aldrin and isodrin are rarely converted into common products by simple chemical action. Derivatives of aldrin are converted into compounds having the skeletal features of the stereoisomeric isodrin series, but with only very low efficiency. Removal of but one of the bridge methylene chlorine atoms in examples of both stereoisomeric hexachlorocyclodienes however results in increased reactivity and skeletal mobility in the presence of electrophilic reagents. For example, both monochloroaldrin and monochloroisodrin are hydrolysed by neat sulphuric acid to give the same tetrachloropentacyclododecanone (I) [or epimer (II) depending on the conditions of purification].



The structures, and mode of formation of ketones (I) and (II) are proposed mainly on the basis of their ¹H nmr spectra and those of their partially deuterated analogues, whose synthesis from deuterated dechloroaldrin derivatives will also be discussed.

66. NMR CHARACTERIZATIONS OF SOME CHLORINATED HYDROCARBON PESTICIDES, THEIR METABOLITES, DERIVATIVES AND COMPLEXES. James D. McKinney, Nancy K. Wilson, Anal. & Syn. Chemistry Branch, National Institute of Environmental Health Sciences, P. O. Box 12233, Research Triangle Park, N. C. 27709, L. H. Keith, A. L. Alford, Southeast Water Laboratory, WQO, EPA, Athens, Ga. 30601.

The use of NMR spectroscopy to more rapidly assess the stereochemistry of some chlorinated polycyclodiene and DDT pesticide metabolites and derivatives will be described. The new shift reagent, Eu(DPM)_3 , was particularly helpful in characterizing the hydroxy compounds normally occurring in aldrin and dieldrin metabolisms, e.g., stereochemical structure proof of the major fecal metabolite of dieldrin in the rat. The utility of chiral lanthanide shift reagents to determine the optical purity of certain pesticide metabolites and ascertain the stereospecificities of certain enzyme systems will be discussed. High resolution NMR studies of DDT-type compounds have revealed that deshielding effects on the *ortho* protons of the aromatic rings are derived from electronegativity of the substituents and the stereochemistry of the molecule. In addition, the NMR spectra of chemical derivatives were very useful in studies aimed at gaining an understanding of the theoretical chemistry of DDT-systems. Complex equilibria involving DDT have also been measured by NMR spectroscopy indicating that two types of complexes are formed.

67. STRUCTURAL APPLICATIONS OF ^{13}C NMR. J.B. Stothers, The University of Western Ontario, Department of Chemistry, London 72, Canada.

The major features of ^{13}C NMR spectra will be briefly reviewed as an introduction to the applications of ^{13}C NMR for structural elucidations. Some examples of structural applications will be described to illustrate the scope of the method and the practical limitations, including some discussion of the instrumentation required. The use of ^{13}C data for stereochemical assignments will also be illustrated. Quantitative applications of ^{13}C NMR will be considered, with particular emphasis on the advantages and disadvantages, and demonstrated with examples of tracer studies employing deuterium-labelled materials. Some comparisons with the more familiar proton techniques will be drawn to illustrate the major advantages of ^{13}C studies.

68. CARBON-13 AND PROTON MAGNETIC RESONANCE STUDIES OF CHLORINATED BIPHENYLS. Nancy K. Wilson and Marshall Anderson, National Institute of Environmental Health Sciences, P. O. Box 12233, Research Triangle Park, N. C. 27709.

Carbon-13 and proton nuclear magnetic resonance spectra have been obtained for a number of chlorinated biphenyls. For some of these compounds, ^{13}C spin-lattice relaxation times have been measured. The spectral parameters are interpreted in terms of the results of CNDO/2 calculations of charge densities and potential energies, molecular motion and equilibrium molecular geometries.

69. APPLICATIONS OF FOURIER TRANSFORM, NMR, AND INFRA-RED SPECTROSCOPY TO PESTICIDES. C. E. Klopfenstein and J. Koskinen, Department of Chemistry, University of Oregon, Eugene, Oregon. 97403.

Time domain techniques for data acquisition from Nuclear Magnetic Resonance and Infrared Instrumentation have made analytical measurements possible that would be very difficult to perform in any other way. Data illustrating the value of computer control in these experiments will be presented using some pesticide compounds as examples. Infrared emission from these compounds will also be discussed.

70. TRACE ANALYSIS BY FOURIER TRANSFORM MAGNETIC RESONANCE: F.H.A. Rummens, Chemistry Department, University of Saskatchewan Regina Campus, Regina, Saskatchewan S4S 0A2 Canada.

ABSTRACT: Pulsed NMR, followed by Fourier Transformation (FT) of the ensuing Free Induction Decay (FID) is a well known technique for boosting the signal-to-noise (S/N) ratio. This method, while originally developed for such "weak" or "insensitive" nuclei like ^{13}C or ^{15}N , is equally applicable to such sensitive and abundant nuclei as ^1H , ^{19}F and ^{31}P . It will be shown that FT NMR, in combination with other signal-improving techniques makes it feasible to observe, identify and determine either small amounts ($1\ \mu\text{g}$) or low concentrations (1 ppm) of certain materials. Detection limits, and their relation to "obtainable signal-to-noise per time unit" will be discussed. Experimental, theoretical and chemical limitations of the various techniques will be shown, using examples mostly from the area of herbicides.

71. IDENTIFICATION OF PHOTOPRODUCTS OF PESTICIDES BY N.M.R. M. J. Zabik, Michigan State University, East Lansing, Michigan. 48823.

Current work indicates that many pesticides are rapidly decomposed by ultraviolet light in the range of 280 to 360 n.m. It is therefore very important to carry out the photoreactions and identify the photoproducts so in assessment of the role that components may or may not play in a sublethal or lethal level in the environment. The use of n.m.r. as an analytical tool for the identification of such photoproducts is indispensable. The discussion will concern the chemical characterization of these photolysis products through the use of n.m.r.

72. NMR STUDIES OF THE BEHAVIOR OF DDT IN MODEL MEMBRANES. R. Haque and I. J. Tinsley, Department of Agricultural Chemistry and Environmental Health Sciences Center, Oregon State University, Corvallis, Oregon 97331.

DDT and related compounds form a complex with lecithin in CCl_4 solution. The proton chemical shifts of DDT-type compounds in the complexes have been discussed in the light of toxicity of such compounds. A method has been developed to obtain a n.m.r. spectrum of DDT in D_2O -lecithin dispersion. The chemical shift data indicate that DDT binds to lecithin in the D_2O -dispersion. The behavior of the metabolite DDE has also been studied in various phospholipids - D_2O dispersions. The results have been explained on the basis of transport of DDE in the bilayers. The addition of various proteins to a D_2O -lecithin-DDT dispersion produced an increase in the line width of the resonance peaks of DDT. This increase in line width indicates binding of DDT to proteins. The possible role of n.m.r. spectroscopy in studying the molecular basis of toxicology of DDT has also been discussed.

73. THE INTERACTION OF BARBITURATES WITH PHOSPHOLIPIDS. T.J. Swift and R.M. Novak, Department of Chemistry, Case Western Reserve University, Cleveland, Ohio 44106.

A hydrogen-bonding interaction between barbiturates and typical phospholipids in non-polar solvents is indicated by proton and ^{13}C NMR measurements on the drugs and by ^{31}P NMR measurements on the phospholipids. In addition the effect of the association is observed on the barbiturate N-H stretch in the infrared spectrum.

The NMR effects of association are relatively large and the stability constants are of the order of 10^2 . It is suggested that the general depressant nature of barbiturates may be accounted for by their association in a similar fashion with a number of other phosphate containing molecules.

FRIDAY MORNING - SYMPOSIUM ON MASS SPECTROMETRY AND NMR OF PESTICIDES - (Concluded) - F. J. Biros and R. Haque, Presiding

74. NMR STUDIES OF LIGOND MACROMOLECULAR INTERACTION. M. A. Raftery, California Institute of Technology, Pasadena, Calif. 91109.

75. IDENTIFICATION OF PESTICIDES IN EFFLUENTS. John M. McGuire, Ann Alford, and Michael Carter, Environmental Protection Agency, Southeast Environmental Research Laboratory, College Station Road, Athens, Georgia 30601.

76. A STUDY OF INTERMOLECULAR COMPLEXES OF BIS (p-CHLOROPHENYL) ACETIC ACID AND SOME BIOLOGICALLY SIGNIFICANT COMPOUNDS. Ralph T. Ross and F. J. Biros, Environmental Protection Agency, Perrine Primate Laboratory, P.O. Box 490, Perrine, Fla. 33157.

Several studies have attempted to relate binding and complexation of pesticides and biological substrates with toxic effects. Nuclear magnetic resonance (NMR) spectroscopic techniques afford a valuable method for determining the structures of organic compounds as well as the interactions of such chemicals with macromolecules. The NMR technique has been used extensively in studies involving drug interactions and has been used to some degree in studies involving pesticide interactions with biological substrates. The chemical interactions of bis(p-chlorophenyl) acetic acid (p,p'-DDA) and bovine serum albumin (BSA) as well as human albumin (HA) have been examined by the NMR technique. Addition of BSA and HA to solutions of p,p'-DDA results in a broadening of

the aromatic protons and α -(methine) proton resonance peaks. Control experiments and studies involving effects of pH, ionic strength, and temperature have demonstrated preferential stabilization of the aromatic rings by selective changes in relaxation rate of the protons attached to the aromatic rings in the presence of BSA and HB. The hydrophobic binding is shown to be inhibited by the competitive binding of *p*-chlorophenyl acetic acid, (*p*-CPA).

77. GC-MS ANALYSIS OF TOXAPHENE RESIDUES. David L. Stalling, Fish-Pesticide Research Laboratory, Bureau of Sport Fisheries and Wildlife, Route #1, Columbia, Missouri 65201.

The isomer composition of toxaphene is not well known. Toxaphene is primarily a mixture of polychlorocamphenes with an average empirical formula of $C_{10}H_{10}Cl_8$. Forty-eight components of toxaphene were separated by GC using a 50 ft. SE-30 S.C.O.T. column (0.020 in. i.d.) and temperature programming. We identified the major ion fragments of toxaphene using a combined double focusing GC-MS-computer system. The mass chromatograms constructed from the combined ion intensities of $m/e = 291 + 293$ in 150 sequential mass scans (taken at 4 second intervals) closely resemble the peak envelope recorded from the total ion monitor. GC peaks in the mass chromatogram determined from $m/e = 83$ (C^+HCl_2) are largest late in the chromatogram while GC peaks containing $m/e = 117$ (C^+Cl_3) generally elute earlier. Approximately 1 μ g. of toxaphene is required for residue confirmation using mass chromatograms constructed from the most intense ion fragments. The spectra are very complex and inferences on isomer structure will be discussed. The use of a SolVent GLC precolumn permits larger injections (25 - 50 μ l.) into the GC-MS, thus concentrating residue extracts of toxaphene.

78. SPIN-LABELING OF LIVING TISSUE - STUDIES OF POLLUTANT-HOST INTERACTION. W.T. Roubal, Northwest Fisheries Center, 2725 Montlake Blvd. E., Seattle, WA 98112.

It has been demonstrated that the spin-labeling technique can be used in studies of pollutant-host interaction in living fish. The present study was aimed at characterizing the mode of transmission and the site of deposition of model compounds to represent constituents of oil spills. Selective probes were used to demonstrate depth of penetration into membranes. Additionally, the effect of short chain hydrocarbons and of aromatics on *in vivo* and *in vitro*-labeled tissue was studied.

79. HAZARDOUS SYNTHETIC ORGANIC COMPOUNDS IN LAKE MICHIGAN FISH. Gilman D. Veith, Douglas W. Kuehl, and Gary E. Glass. U. S. Environmental Protection Agency, National Water Quality Laboratory, 6201 Congdon Boulevard, Duluth, Minnesota 55804.

The presence of persistent hazardous chemicals in Lake Michigan at levels which are greater than those thought to be safe for human consumption has placed both the commercial and sport fisheries of this lake in jeopardy. Due to the variety of synthetic chemicals in Lake Michigan, chemical characterizations of the chemicals have been conducted using liquid chromatography and GLC/mass spectrometry. This paper will discuss the use of the mass spectrometry facilities at the National Water Quality Laboratory. Results have shown that the major hazardous chemicals in the Lake Michigan fish include agricultural pesticides, phthalate esters and chlorinated biphenyl mixtures (PCBs). Analyses of more than 600 fish have shown that the mean concentration of PCBs (predominately Aroclor 1254) ranged from 2 μ g/gm (whole tissue) in smelt to 14 μ g/gm in salmon and trout.

80. NMR STUDIES OF PREFERENTIALLY ORIENTED WATER AT INTERFACES. D. E. Woessner, Mobil Research and Development Corporation, Field Research Laboratory, Dallas, Texas 75221.

Molecular interactions allow surfaces or interfaces to restrict the possible orientations of water molecules. It is shown how these preferentially oriented water molecules can give rise to a doublet in the NMR spectrum. Such doublet spectra are observed for water in contact with a number of different organic and inorganic systems. The results of proton and deuterium pulsed NMR measurements on oriented sodium hectorite clay suggest that the observed doublet splitting results from preferential orientation of the first several molecular layer of water near the clay surface. A method is described for determining the doublet splitting constant of a sample made by mixing water and a

powder. Details of clay lattice substitution, the cation exchange capacity, and the identity of the exchangeable cation have only minor effects on the deuteron doublet splitting for samples containing montmorillonite type clays with more than several monolayers of water. The results of measurements on illite and kaolinite are consistent with preferential orientation similar to that for montmorillonite. Comparison of doublet splitting phenomena for sodium hectorite clay, collagen, lithium-DNA, and rayon suggests that this statement might be extended to some organic substrates.

81. DETERMINATION OF METASTABLE TRANSITIONS IN THE MASS SPECTRA OF PESTICIDES BY DIRECT ANALYSIS OF DAUGHTER IONS. F. J. Biros and J. F. Ryan III, Environmental Protection Agency, Perrine Primate Lab., P.O. Box 490, Perrine, Florida 33157.

Metastable transitions are useful in elucidating ion decomposition pathways of organic compounds. In many instances involving identifications of unknown compounds, e.g., pesticide metabolites and environmental degradation products, these interpretations are of great value in establishing the structural identity of the material. Conventional defocusing techniques and ion kinetic energy spectroscopy are two methods of detection of metastable transitions in the first field-free region of the mass spectrometer. In double focusing mass spectrometers with reversed field arrangements, a third technique called direct analysis of daughter ions (DADI), becomes possible which permits analysis of these transitions in the second field-free region of the mass spectrometer. Information will be presented describing the application of this latter technique to the detection of metastable transitions in the mass spectra of a group of organophosphate and carbamate pesticides. Complex ion decompositions involving unique rearrangement reactions were observed in many cases. The molecular ion of Abate, e.g., at m/e 466, decomposes into approximately fourteen distinct ion species by a variety of mechanisms. Metastable ion spectra obtained by the DADI technique for major fragment ions as well as the molecular ion in each compound are discussed with reference to the structural characteristics of the parent molecule.

82. NMR OF ADSORBED MOLECULES WITH A VIEW TOWARD PESTICIDE CHEMISTRY. Henry A. Resing, and J. Thompson, Surface Chemistry Branch Code 6170, Chemistry Division, Naval Research Laboratory, Washington, D.C. 20390.

This paper describes how the nuclear magnetic resonance relaxation times yield for certain adsorbed molecules such things as (a) surface diffusion coefficients, (b) rotational jump times, (c) distributions of binding energies, and (d) kinetics of phase exchange. There is sufficient data to conclude, for instance, that adsorbed water molecules on inorganic and biological materials possess a mobility much closer to that of liquid water than to that of ice. Recent measurements of chemical shift tensors for adsorbed molecules (a) show that electronic structures for physisorbed molecules are not greatly perturbed, as well as (b) confirm the motional picture obtained from relaxation times.

DDT dehydrochlorinase

MW 120000, tetramer

binds to substrate by hydrophobic bonding

microsomal oxidases

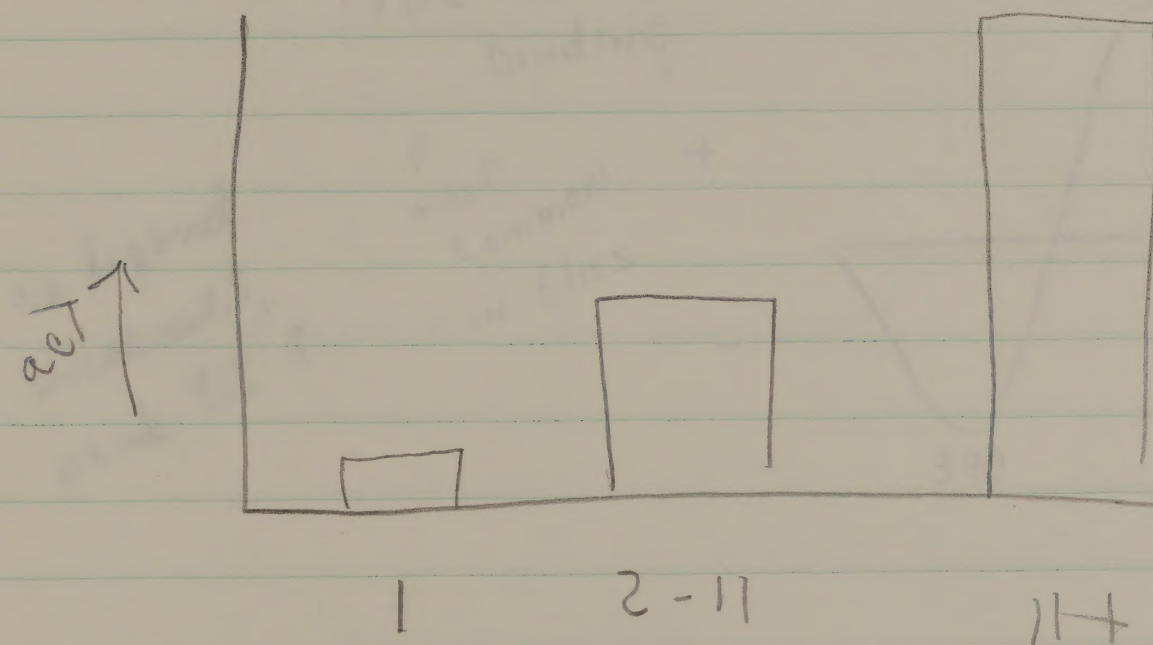
regulated by

DDT induces mRNA synthesis

by three RNA polymerase

Send WARREN DWANE copy of revised 15647 MS

Rhee
Reed - Ho



Host plants

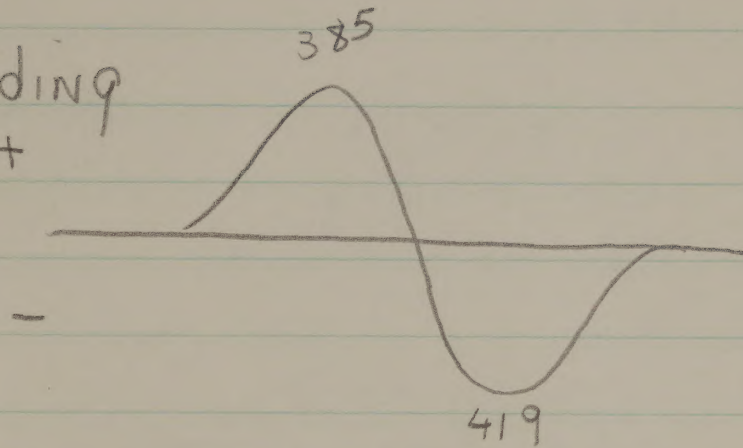
dihydroisodrin

~~no~~ oxidase activity in vivo but not in vitro in pupae

P-450

Type I binding

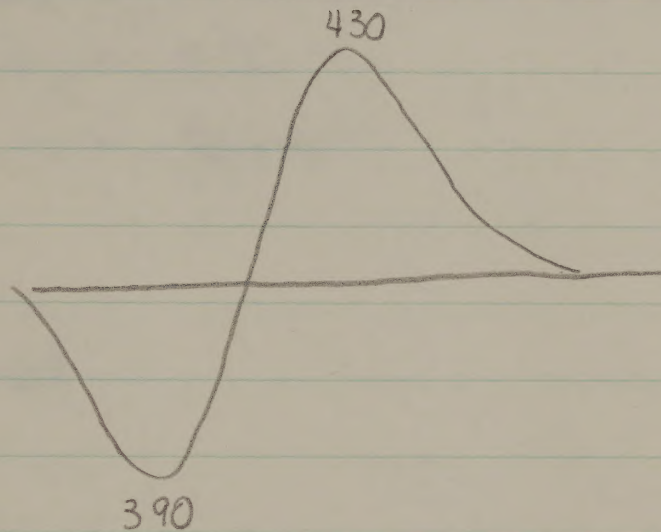
+



Type II binding

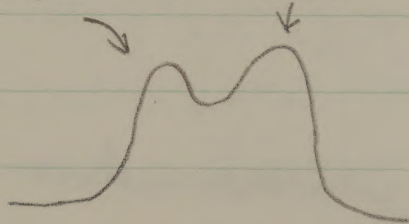
↑ most common in flies

+

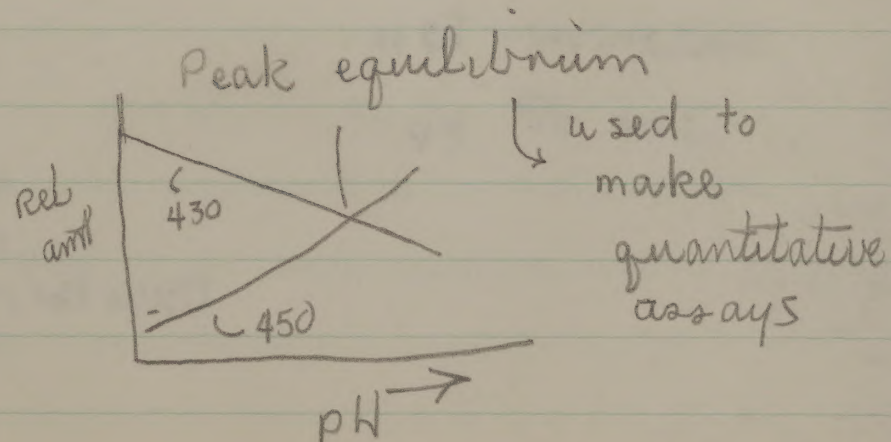


as ligand increases, peak ht ↑

430 & 450



biphenyl (caused by Δ pH)



Heredity of Cyt P-450

Reasons for higher rate of metabolism in R insects

- decreased enzyme degradation
- mutant enzyme is more efficient
- more enzyme

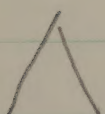
Regulation may be involved

- 1 - hormones
- 2 - increased mRNA
- 3 - movement of mRNA from nuc to cyto
- 4 - transcription

Gene redundancy

S
mo mo

R



|| mo
|| mo
~~normal~~ induced

Net increase
vs % inc.

Q3A-7 to 7/1/2007

Section 2 - Distribution of star 19/1/2007
metabolic amino acid
in the liver and kidney
in the liver and kidney

Section 3 - Distribution of star 19/1/2007
in the liver and kidney

Section 4 - Distribution of star 19/1/2007
in the liver and kidney

Section 5 - Distribution of star 19/1/2007
in the liver and kidney

Section 6 - Distribution of star 19/1/2007
in the liver and kidney

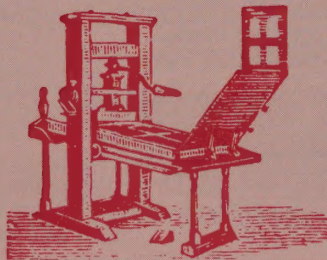
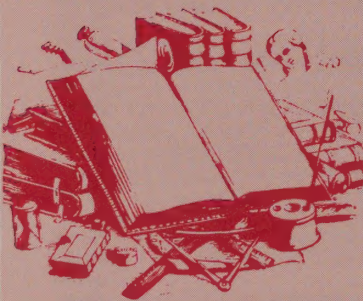
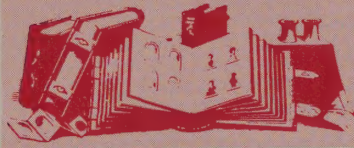
phenobarbital in diet

0.5% $\rightarrow$ 2.0%

phenobarbital is -metabolism by
mfo's

Sci.
Meyer & MARVER 191:64

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C. H. Van Middlelem, Presiding

- 9:00 - Introductory Remarks.
- 9:05 - 1. Degradation of Ethylenethiourea in Soil. D. D. Kaufman,
C. L. Fletcher.
- 9:20 - 2. CYTROLANE® Systemic Insecticide: Metabolism in Cotton
Plants. J. Zulalian.
- 9:40 - 3. The Effect of Isopropyl-3-Chlorocarbanilate and Its Plant
Metabolites Upon Mitochondrial Oxidative Phosphorylation.
G. G. Still, D. G. Rusness.
- 9:55 - 4. Mass Spectral Characterization of the Sheep Urinary Metabolites
from Rulene. J. E. Bakke, C. E. Price.
- 10:10 - 5. Synthesis of Glucosides of Isopropyl 3-Chlorocarbanilate
Metabolites. G. G. Ecke.
- 10:25 - 6. Correlations of Toxicity and Acetylcholinesterase (AChE) Inhibition
in 2-Alkyl Substituted 1,3-Benzodioxolyl-4 N-Methylcarbamates and
Related Compounds. C. C. Yu.
- 10:40 - 7. Isolation of Acylanilide-Hydrolyzing Microbial Enzymes. J. Blake,
D. D. Kaufman.
- 10:55 - 8. The Properties of An Aryl Acylamidase From Dandelion Which
Hydrolyzes 3',4'-Dichloropropionanilide. R. E. Hoagland,
E. D. Handel, G. Graf.
- 11:10 - 9. Photolysis of 2,2-Dichloropropionic Acid in Aqueous Solution.
F. S. Tanaka, R. G. Wien.

- 11:25 - 10. Metabolism, Excretion and Storage of Hexachlorobenzene
in The Male Rat. H. M. Mehendale, H. B. Matthews.
- 11:40 - 11. The Persistence of Dieldrin and Heptachlor in a Field Soil.
H. P. Freeman, A. W. Taylor, W. M. Edwards.

MONDAY AFTERNOON

Panel Discussion: PR70-15 Environmental Impact Requirements

R. A. Conkin, Presiding

2:00 - Introductory Remarks.

2:10 - 12. Environmental Aspects of Pesticide Registration. C. W. Collier.

2:40 - 13. A Proposed Approach to PR 70-15 Guidelines For Studies To
Determine The Impact of Pesticides On the Environment. C. A. I.
Goring, J. W. Hamaker, D. A. Laskowski.

3:00 - Discussion.

3:10 - 14. One Industrial View of Notice PR 70-15. J. E. Boyd.

3:30 - 15. An Academic Look at Environmental Regulatory Requirements.
R. L. Metcalf.

4:00 - Audience Participation

TUESDAY MORNING

Section A

Symposium on New Aspects of Organophosphorus Pesticides.

T. R. Fukuto, Presiding

- 9:00 - Introductory Remarks.
- 9:10 - 16. Oxidative Rearrangement of Organophosphorus Thionate Esters.
 D. A. Wustner, T. R. Fukuto, M. A. H. Fahmy.
- 9:35 - 17. Newer Aspects of Metabolism of Phosphonate Insecticides.
 J. J. Menn, J. B. McBain.
- 10:05 - Discussion.
- 10:10 - 18. Organophosphorus Insect Chemosterilants. A. B. Borkovec.
- 10:50 - 19. Structure and Bioactivity of Orthene Analogs. P. S. Magee.
- 11:20 - Discussion.
- 11:25 - 20. Structure and Fungitoxicity of Organophosphorus Compounds.
 H. Tolkmith, D. Mussell.
- 11:55 - Discussion.

TUESDAY MORNING

Section B.

General

H. F. Enos, Presiding

- 9:00 - Introductory Remarks.
- 9:05 - 21. Vapor-Phase Photodecomposition of p,p'-DDT and Its Relatives.
 K. W. Moilanen, D. G. Crosby.
- 9:25 - 22. Investigation For Bound Residues in Tissues From Cattle Fed 2,4,5-T.
 D. J. Jensen, P. W. Miller, L. F. Berhenke.
- 9:40 - 23. Detection of Mammalian Exposure to Carbamate Insecticides.
 H. C. Sullivan, T. Shafik.
- 9:55 - 24. Determination of Metaldehyde in Crop Tissue.
 S. Selim, J. Seiber.
- 10:15 - 25. Development of Methods Using 1-Fluoro-2,4-Dinitrobenzene for
 Gas-Liquid Chromatography of Methomyl. C. E. Mendoza,
 J. B. Shields.
- 10:30 - 26. Direct Gas Chromatographic Analysis of Methomyl Residues in Soil,
 Sediment, Water and Tobacco Utilizing the Flame Photometric
 Detector. R. G. Reeves, D. W. Woodham.
- 10:40 - 27. Residues of Dimethoate and Its Oxygen Analog On and In Citrus Leaves
 Following a Helicopter Treatment of The Trees with a Dimethoate ULV
 Concentrate and High Volume Spray. D. W. Woodham, R. G. Reeves,
 C. B. Williams, H. Richardson, C. A. Bond.
- 10:50 - 28. Esters of Sulfonyl Chlorides as Derivatives for the Gas Chromato-
 graphic Analysis of Carbamate Pesticides. H. A. Moyer.

- 11:10 - 29. Response of the Electrolytic Conductivity Detector in the Oxidative Mode to Herbicides and Its Use in Residue Analysis in Soil and Water by Gas Chromatography. R. Purkayastha.
- 11:20 - 30. Theory of Controlled Release--Diffusion. R. L. Collins, S. Doglia.
- 11:35 - 31. Acaricidal Enol Esters of 2-Aryl-1,3-Indandiones, Structure-Activity Relationships. J. A. Durden, A. A. Sousa, J. F. Stephen.

TUESDAY AFTERNOON _____

2:00 - 32. International Pesticide Chemistry Research Award.

Address - Selective Herbicidal Activity. R. L. Wain.

Symposium on New Aspects of Organophosphorus Pesticides.

T. R. Fukuto, Presiding

3:05 - 33. Bensulide, S-(O,O-Diisopropyl Phosphorodithioate) of N-(2-Mercapto Ethyl) Benzene Sulfonamide. L. W. Fancher, R. A. Simone, E. G. Teach.

3:25 - 34. Thiophosphorylimino Oxazolidines, Thiazolidines, and Related Compounds as Insecticides. E. J. Gaughan, J. J. Menn.

3:50 - 35. Metabolism, Biochemical and Biological Aspects of Nema~~cur~~ and Related Phosphoramidate Compounds. T. B. Waggoner, A. M. Khasawinah.

4:30 - (36) Some Recent Advances in the Study of Organophosphate Metabolism. R. M. Hollingworth.

WEDNESDAY MORNING

Symposium on Biochemistry of Insect Resistance

F. W. Plapp, Jr., Presiding

- 9:00 - Introductory Remarks.
- 9:10 - 37. Biochemistry of Insect Resistance to DDT. M. Agosin.
- 9:50 - 38. Genetic Studies on Factors Affecting Cross-Resistance Spectra
 In Insecticide-Resistant Insects. R. M. Sawicki.
- 10:30 - 39. Biochemistry of Insect Resistance. Non-Microsomal Mechanisms.
 N. Motoyama, W. C. Dauterman.
- 11:05 - 40. Insecticide Resistance Factors in Lepidopterous Larvae.
 R. I. Krieger.
- 11:40 - Discussion.

WEDNESDAY AFTERNOON

Section A

Symposium on Biochemistry of Insect Resistance

F. W. Plapp, Jr., Presiding

- 2:00 - Introductory Remarks.
- 2:10 - 41. Third Generation Pesticides: The Potential For The Development
 of Resistance by Insects. S. B. Vinson.
- 2:40 - 42. Microsomal Cytochrome P-450: Characterization and Possible Role in
 Insecticide Resistance in Musca Domestica. E. Hodgson, L. G. Tate,
 A. P. Kulkarni, F. W. Plapp, Jr.
- 3:10 - 43. The Induction of Detoxifying Enzymes in Insects. L. C. Terriere.
- 3:55 - 44. Evidence For a Key Role of d-Aminolevulinic Acid Synthetase in Insecti-
 cide Resistance. F. W. Plapp, Jr.
- 4:40 - Discussion.

WEDNESDAY AFTERNOON

Section B

General

R. D. Moss, Presiding

- 2:00 - Introductory Remarks.
- 2:05 - 45. Effect of Structure on the Reactivity of Dimethyl Substituted-
Phenyl Phosphates With Nucleophilic Reagents. D. W. Osborne,
V. Stevens.
- 2:20 - 46. Synthesis and Biological Activity of a New Class of Oxime
Carbamates. T. A. Magee, L. E. Limpel.
- 2:35 - 47. Tetrahydrobenzothiazole Herbicides - I. J. E. Engelhart.
- 2:50 - 48. Tetrahydrobenzothiazole Herbicides - II. J. E. Engelhart.
- 3:05 - 49. PCB Content of Selected Health Foods. W. B. Wheeler,
J. A. Koburger, H. Appledorf.
- 3:20 - 50. Retention and Excretion of Polychlorinated Biphenyl Residues by
Laying Hens. G. F. Fries, R. J. Lillie, H. C. Cecil, J. Bitman.
- 3:35 - 51. Effects of a Single Oral Injection of Polychlorinated Biphenyls and
Terphenyls and a Polybrominated Biphenyl on Pentobarbital
Sleeping Times of Japanese Quail. H. C. Cecil, S. J. Harris,
J. Bitman.
- 3:50 - 52. Retention and Excretion of Polybrominated Biphenyls by Hens and
Cows. G. F. Fries, L. W. Smith, H. C. Cecil, J. Bitman, R. J. Lillie.
- 4:05 - 53. Polynitro-Substituted Arylsulfon-1-Naphthalides as Chemosterilants
For House Flies. A. B. DeMilo, A. B. Bořkovec.
- 4:20 - 54. Electron Induced Fragmentation of Conjugated Dienoate Esters.
L. L. Dunham, J. W. Young, C. A. Henrick.

4:40 - 55. Electron Capture-Gas Liquid Chromatography of 2,6-Dichloro-4-Nitroaniline and Its Metabolite in Tissues and Excreta.

R. F. Moseman.

THURSDAY MORNING

Symposium on Mass Spectrometry and NMR of Pesticides

F. J. Biros, Presiding

- 9:00 - Introductory Remarks.
- 9:05 - 56. Characterization of Chlorinated Hydrocarbon Pesticide
Metabolites of the DDT and Polycyclodiene Types By
Electron Impact and Chemical Ionization Mass Spectrometry.
J. D. McKinney, E. O. Oswald.
- 9:25 - 57. The Ion Kinetic Energy Spectra of Pesticides and Related
Compounds. S. H. Safe, O. Hutzinger, W. D. Jamieson.
- 9:45 - 58. Self-Training Interpretive and Retrieval System for Mass
Spectra. K. S. Kwok, R. Venkataraghavan, G. Pesyna, I.
Sakai, J. W. Serum, A. Tatematsu, R. G. Werth, F. W. McLafferty.
- 10:05 - 59. Positive and Negative Chemical Ionization Mass Spectra of Polychlorinated
Pesticides. R. C. Dougherty, J. D. Robert, F. J. Biros.
- 10:25 - 60. Mass Spectra of Pesticide Photoproducts. J. D. Rosen.
- 10:45 - 61. Electron Impact Mass Spectra of Several Aromatic Ring Systems.
A. Curley, R. W. Jennings, V. W. Burse, E. Villanueva.
- 11:05 - 62. Mass Spectral Analysis of Some Carbanilate Herbicides and Their Aryl
Hydroxylated Metabolites. G. G. Still.
- 11:25 - 63. Identification of Insecticide Photoproducts by Mass Spectrometry.
B. R. Layton, E. G. Alley.
- 11:45 - 64. The Application of Mass Spectrometry to the Study of Nitroaniline Derived
Herbicides. J. R. Plimmer, U. I. Klingebiel.

THURSDAY AFTERNOON

Symposium on Mass Spectrometry and NMR of Pesticides

R. Haque, Presiding

2:00 - Introductory Remarks.

2:05 - 65. ^1H NMR Spectra and Structure of a Unique Saturated Tetra-chloropentacyclododecanone Derived From Monodechloroaldrin and Monodechloroisodrin. K. Mackenzie, C. H. M. Adams, D. J. Cawley.

2:25 - 66. NMR Characterizations of Some Chlorinated Hydrocarbon Pesticides, Their Metabolites, Derivatives and Complexes. J. D. McKinney, N. K. Wilson, L. H. Keith, A. L. Alford

2:45 - 67. Structural Applications of ^{13}C NMR. J. B. Stothers.

3:05 - 68. Carbon-13 and Proton Magnetic Resonance Studies of Chlorinated Biphenyls. N. K. Wilson, M. Anderson.

3:25 - 69. Applications of Fourier Transform, NMR, and Infra-Red Spectroscopy to Pesticides. C. E. Klopfenstein, J. Koskinen.

3:45 - 70. Trace Analysis by Fourier Transform Magnetic Resonance. F. H. A. Rummens.

4:05 - 71. Identification of Photoproducts of Pesticides by NMR. M. J. Zabik.

4:25 - 72. NMR Studies of the Behavior of DDT in Model Membranes. R. Haque, I. J. Tinsley.

4:45 - 73. The Interaction of Barbiturates With Phospholipids. R. M. Novak, T. J. Swift.

FRIDAY MORNING

Symposium on Mass Spectrometry and NMR of Pesticides

F. J. Biros and R. Haque, Presiding

- 9:00 - Introductory Remarks.
- 9:05 - 74. NMR Studies of Ligand Macromolecular Interaction. M. A. Raftery.
- 9:25 - 75. Identification of Pesticides in Effluents. J. M. McGuire,
A. Alford, M. Carter.
- 9:45 - 76. A Study of Intermolecular Complexes of Bis (p-Chlorophenyl) Acetic
Acid and Some Biologically Significant Compounds. R. T. Ross,
F. J. Biros.
- 10:05 - 77. GC-MS Analysis of Toxaphene Residues. D. L. Stalling.
- 10:25 - 78. Spin-Labeling of Living Tissue - Studies of Pollutant-Host Interaction.
W. T. Roubal.
- 10:45 - 79. Hazardous Synthetic Organic Compounds in Lake Michigan Fish.
G. D. Veith, D. W. Kuehl, G. E. Glass.
- 11:05 - 80. NMR Studies of Preferentially Oriented Water at Interfaces.
D. E. Woessner.
- 11:25 - 81. Determination of Metastable Transitions In The Mass Spectra of
Pesticides By Direct Analysis of Daughter Ions. F. J. Biros,
J. F. Ryan, III.
- 11:45 - 82. NMR of Adsorbed Molecules With a View Toward Pesticide Chemistry.
H. A. Resing, J. Thompson.

Whitten
8-27

"MECHANISMS OF SURVIVAL IN TOXIC ENVIRONMENTS"

A Symposium Organized by the

~~American Society of Zoologists~~
(Division of Comparative Physiology and Biochemistry)

December 28, 29 and 30, 1973

Rice Hotel, Houston, Texas

MECHANISMS OF SURVIVAL IN TOXIC ENVIRONMENTS

Symposium Chairman: M.A.Q. Khan

SESSION I

December 28

- 9:00 A.M. Introduction: E.B. Hadley, University of Illinois, Chicago Circle
Impact of Chemical Pollutants on the Biology of
Organisms. R. L. Rudd, Chairman
- 9:10 A.M. Ecosystem Studies with Pesticides and Other Chemical
Pollutants. R. L. Rudd, R. B. Craig and R. L. Garrett,
Department of Zoology, University of California, Davis,
California.
- 9:50 A.M. Environmental Impact of Pesticides. D. Pimentel,
Department of Entomology and Section of Ecology and
Systematics, Cornell University, Ithaca, New York.
- 10:30 A.M. The Problem of Lead Poisoning. R. A. Kehoe, Kettering
Laboratory, University of Cincinnati, Cincinnati, Ohio.
- 11:00 A.M. Environmental Pollution in Relation to Estuarine Birds.
H. M. Ohlendorf, Patuxent Wildlife Research Center,
U. S. Department of Interior, Laurel, Maryland.

SESSION II

Detoxication as Mechanisms of Survival. R. H. Adamson, Chairman

- 1:30 P.M. Detoxication of Insecticides in Microorganisms.
F. Matsumura, Department of Entomology, University
of Wisconsin, Madison, Wisconsin.
- 2:00 P.M. Detoxication of Acaricides in Animals. C. O. Knowles,
Department of Entomology, University of Missouri,
Columbia, Missouri.
- Break 15 min.
- 2:45 P.M. Comparative Metabolism of Foreign Compounds in Fishes.
R. H. Adamson, Laboratory of Chemical Pharmacology,
National Cancer Institute, N. I. H., Bethesda, Maryland.
- 3:40 P.M. Detoxication of Foreign Chemicals by Invertebrates.
M. A. Q. Khan, Department of Biological Sciences,
University of Illinois at Chicago Circle, Chicago, Illinois.
- 4:10 P.M. Detoxication of Insecticides: Comparative Approach.
J. N. Smith, Department of Zoology, Victoria University,
Wellington, New Zealand.

SESSION III

December 29

Role of Mixed-Function Oxidase in Survival in Toxic Environments.
E. Hodgson, Chairman

- 9:00 A.M. Mixed-function Oxidase in the Metabolism of Foreign Compounds. R. W. Estabrook, Department of Biochemistry, Southwestern Medical School at Dallas, The University of Texas, Dallas, Texas.
- 9:40 A.M. Comparative Studies of the Cytochrome P-450 and its Interaction with Pesticides. E. Hodgson, North Carolina State University, Raleigh, North Carolina.
- 10:30 A.M. Genetic Regulation and Mechanisms of P-450 Oxygenase Action. I. C. Gunsalus and V. P. Marshall, University of Illinois, Urbana, Illinois.
- 11:10 A.M. Mammalian Mixed-function Oxidase: Mechanism of Induction and Role in Carcinogenesis. F. J. Wiebel and H. E. Gelboin, Chemistry Branch, National Cancer Institute, N. I. H., Bethesda, Maryland.

SESSION IV

Recent Advances in Insecticide Research. F. W. Plapp, Jr., Chairman

- 1:00 P.M. Insecticide Resistance in Vertebrates. J. Yarborough, Department of Zoology, Mississippi State University, State College, Mississippi.
- 2:10 P.M. Insecticide Resistance in Insects and Its Ecological and Economic Thrust. A. S. Perry, Research Laboratories, U. S. D. H. E. W., Savannah, Georgia.
- 2:50 P.M. Genetics of Detoxication Systems in Insects. F. W. Plapp, Jr., Texas A. & M. University, College Station, Texas.
- 3:40 P.M. Photolysis of Insecticides and It's Ecological Significance. D. J. Sutherland, J. D. Rosen and M. A. Q. Khan, Rutgers University - The State University of New Jersey, New Brunswick, New Jersey and University of Illinois at Chicago Circle, Chicago, Illinois.

SESSION VDecember 30

Non-pesticidal pollutants. J. P. Bederka, Jr., Chairman

- 9:00 A.M. Factors Affecting the Behavior of Chemicals in Environment. R. Haque, Department of Agricultural Chemistry, Oregon State University, Corvallis, Oregon.
- 9:40 A.M. Ecological and Health Effects of Pollutants in Automobile Exhaust. D. G. Penney, J. P. Bederka, Jr., Z. A. Saleem and W. F. Coello, University of Illinois at Chicago Circle and Medical Center, Chicago, Illinois.
- 10:30 A.M. Physiological Effects of Sublethal Concentrations of Oil, Heavy Metals and PCB's on Estuarine Organisms. J. W. Anderson, J. M. Neff and S. R. Petrocelli, Department of Biological Sciences, Texas A & M University, College Station, Texas.
- 11:10 A.M. Biodegradation of Detergent Builders and Other Synthetic Organic Chemicals. C. Warren, Monsanto Chem., Co., St. Louis, Missouri.
- 11:30 A.M. Costs and Benefits of Pesticides, G. C. Decker, Emeritus Chief, Section of Economic Entomology, Illinois Natural History Survey, Urbana, Illinois.

